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DEVELOPMENT OF A HIGH-PURITY FILTER FOR HIGH TEMPERATURE PARTICULATE SAMPLING AND ANALYSIS

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Arthur D. Little, Incorporated

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DEVELOPMENT OF A HIGH-PURITY FILTER FOR HIGH TEMPERATURE PARTICULATE SAMPLING AND ANALYSIS

by

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FOREWORD

The work described in this report was monitored for the Environmental Protection Agency by Mr. John Davis and Dr. Kenneth Knapp.

The authors are indebted to all program participants for their cooperation and effort, but especially to Dr. D. W. Lee, Dr. E. T. Peters, and Mr. W. J. Smith (consultant) for their advice and inspiration.

Emission spectrographic analyses were performed by Kent Laboratories of Waltham, Massachusetts. X-ray fluorescence analyses were performed by Columbia Scientific Industries, Inc., of Austin, Texas, and the sponsor. Neutron activation analyses were performed by the sponsor.

This report has been reviewed by the Environmental Protection Agency and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Agency, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

Arthur D Little Inc

SUMMARY

The purpose of this program has been to develop and characterize a high-purity filter medium for measuring particulates in stack gases at 230 to 540°C. Sufficient purity to allow optimum use of the detection limits of atomic absorption (AAS) and flame emission spectrometry (FES), and reproducible collection efficiency of 99% for dense particles as small as 0.05 μm were primary objectives.

The work has included analysis and purification of filter materials, filter handsheet preparation, and filter characterization. The latter includes the determination of filter suitability for use in X-ray fluorescence and neutron activation analysis of collected particulates.

Johns-Manville Co. 99.2% silica fibers have been made into handsheet filters with satisfactory efficiency, temperature resistance, cost (about $\$1/ft^2$), strength (about 1 lb/in.), and flexibility. The strength and flexibility result from a novel combination alkaline and 800°C heat treatment. The heat treatment also decreases extractable impurities and moisture sensitivity. The application of this treatment to machine production of filter media should be evaluated.

Almost all purity requirements for optimum AAS and FES analysis have been achieved. Major extractable impurities in excess of AAS and FES detection limits are lead, iron, aluminum, chromium, titanium, and zinc. The filters appear highly promising for X-ray fluorescence analysis but should be further evaluated. Suitability of the filters for neutron activation analysis is uncertain but should also be further evaluated. Higher purity silica fibers and other similar fibers have been developed by several companies recently (but not in time to be evaluated in this program). These fibers should be evaluated.

Collection efficiency has been measured with liquid dioctyl phthalate (DOP) particles of 0.3 μm diameter. Efficiencies of 99 to 99.99% have been achieved by using fibers of various diameters. Efficiency of collection of solid, high-density particles has not been measured, but we expect the 99.9% DOP efficiency filters to be at least 99% efficient on such particles.

The strengthened filters are insensitive to humidity (similar to Gelman A glass filters), insoluble in most acids and organic solvents, and slightly alkaline (pH 9 to 10).

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DEVELOPMENT OF A HIGH-PURITY FILTER FOR HIGH TEMPERATURE PARTICULATE SAMPLING AND ANALYSIS

I. INTRODUCTION

As a result of recent improvements in trace metal analytical techniques, sources of many potentially hazardous chemicals in the atmosphere can be located by sampling and analysis of stack gas particulates. However, strong, high-purity filter media are not available for high-temperature use (230 to 540°C). Furthermore, the efficiency of filter media in current use under some conditions is suspected to be inadequate.

The primary purpose of this program was to develop a suitable filter, as follows:

1. Sufficient purity to allow optimum use of the detection limits of atomic absorption and flame emission spectrophotometric analysis for Ag, Al, As, B, Ba, Be, Cd, Ca, Co, Cr, Cu, Fe, Hg, Hf, In, Mn, Mo, Ni, P, Pb, Sb, Sn, Ta, Te, Ti, V, W, Zn, Rh, Mg, K, and Zr.

2. Reproducible collection efficiency of 99% for spherical particles of 5 g/cc minimum density and 0.05 μm minimum diameter at a face velocity of 100 cm/sec.

3. Sufficient strength to resist fiber "blowoff," tearing, cracking, etc.

4. Reasonable cost.

A secondary purpose was to determine the following filter characteristics:

1. Applicability of filter for in-situ X-ray fluorescence and neutron activation analyses.

2. Effect of humidity on filter weight.

3. Solubility in acid, alkaline, and organic media.

The work has consisted of review and planning studies, analysis and purification of candidate filter materials, filter handsheet development, and filter characterization. These activities are discussed in the following sections.

II. REVIEW AND PLANNING

A. STACK GAS SAMPLING FOR PARTICULATES

Stack sampling can be an extremely difficult task with severe constraints on materials and equipment. One of the most obvious constraints is high temperature. Some additional factors of relevance to this filter development program are as follows:

1. Particulate loadings can be highly variable. At very low loadings, high-velocity sampling with high-purity filters is necessary for trace-level analytical work so that excessive sampling times can be avoided. At high loadings, high filter capacity for particulates (resistance to plugging) is important.

2. Stack gases often contain gases that corrode or adsorb on filters, resulting in particulate measurement errors.

3. Stack access is commonly limited to 3" diameter sampling ports. When the sample must be obtained inside the stack, the choice of filter type and size is very limited.

Sampling stacks for particulate emissions is similar in many ways to many other particulate sampling tasks. Filters of high purity, high efficiency, high weight stability, low flow resistance, adequate capacity, and consistent uniformity are commonly needed but not available. In addition, however, stack sampling filters require resistance to temperatures of 230 to 540°C.

Most particulate sampling filters for ambient temperatures have been discussed in references 1 to 3. Relatively recent additions to these available filters are GE Nuclepore membranes and Selas silver membranes. Filters currently used for high-temperature stack sampling and their limitations are as follows:

- Teflon Membranes - Temperature resistance is limited to about 260°C. Flow resistance is high.

- Porous Ceramics[4] - (e.g., Norton alundum thimbles). Flow resistance is high, and the necessary purity is expected to be difficult to achieve.

- Selas Silver Membranes - Flow resistance and reactivity are high. Membrane weight change is expected to result from reaction with H_2S and acid gases.

- High-Efficiency Glass Fiber Filters - Purity is inadequate but this filter is useful to about 540°C and is specified for use in the EPA stack sampling train. The commercial suppliers and performance characteristics of this filter are discussed in the Appendix.

B. SILICA FIBER APPROACH

High-efficiency silica fiber filter material can be obtained commercially from Pallflex Products Corporation ("Tissuquartz") for $1.00/ft^2 or less. It is made on a stainless steel Fourdrinier type of papermaking machine with Johns-Manville 98.5% "Microquartz" fibers. The material is weak and too moisture-sensitive for gravimetric measurements, but suitable heat treatment and/or chemical treatment should eliminate these problems. Because "Microquartz" is prepared by leaching blown glass fibers, residual extractable impurities are very low. Heat treatment to evaporate volatile metals and close the porous leached structure is expected to further decrease extractable impurities. Purity should also be increased by using the following higher purity fibers:

1. General Electric Co. 10 μm Drawn, Fused Quartz Fibers

These fibers are reported to be the strongest and highest purity (50 to 100 ppm total impurities) commercially available quartz fibers, but continued availability is somewhat uncertain. Price is about $100/lb. GE states that 10 to 15 impurities are present, but that the major ones are Al and Na. This fiber is available from GE in continuous lengths only and will require cutting to half-inch lengths or less for filter preparation.

2. H. P. Stevens & Co. 7 to 9 μm Diameter Drawn, Fused Quartz Fibers (Astroquartz)

The standard purity quoted for this material is 99.95% silica, but up to 99.98% purity lots can be specified. Fibers are chopped to various lengths, including 1/4" and 1/2". Price is $35 to $75/lb.

3. J. P. Stevens & Co. Blown, Fused Quartz Wool (Astroquartz)

These fibers are similar in purity to the chopped, drawn Astroquartz. They are crimped and vary in size from 1 to 12 μm diameter and 1 to 2-1/2" long. It will be necessary to chop these fibers to a maximum length of 1/2" and possibly as short as 1/8" to permit proper dispersion and sheet formation. Price is $40 to $75/lb.

4. Johns-Manville 99.2% to 99.5% "Microquartz"

(0.5 to 0.7 μm diameter and 0.7 to 1.6 μm diameter in unspecified lengths.)

Because "Microquartz" is the best currently available fiber for making the desired filters and because "Microquartz" development work is still in progress, we will identify all currently available grades in terms of process conditions, as follows:

a. 98.5% "Microquartz" - Made with one leach in "old process"* equipment at 190°F with technical grade H_2SO_4 and tap wash water. Price $4 to $8/lb.

b. 99.2% "Microquartz" - Made with one leach in "old process"* equipment at 190°F with technical grade H_2SO_4 and deionized wash water. Price $15 to $35/lb.

c. 99.5% (best effort) "Microquartz" - Made with two leaches in "new process"* equipment at 190°F with reagent grade H_2SO_4 and deionized wash water. Johns-Manville will aim for, but not guarantee, 99.5%. Actual analyses can be obtained. Availability and price of production quantities is uncertain.

High-efficiency glass fiber filters and silica fiber filters are made commercially by conventional papermaking processes. The same processes should be capable of making high-purity filters, provided that:

1. Raw materials (including water and drying air) are high purity.

2. Equipment is clean (preferably stainless steel).

3. Housekeeping is immaculate. Work area must be kept clean and free of contaminants.

Because silica fiber filters have potential for meeting the program objectives, they were selected for further study. We also considered other high-temperature fibers[5,6] but find them less attractive than silica. For example, boron nitride (Carborundum Co.) is expensive ($350 to $600/lb), only 99% pure, and would not permit boron analyses. Graphite could gain or lose weight by gas adsorption or oxidation. Organic fibers such as Kynol II (phenolic) have inadequate temperature resistance.

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*Johns-Manville is developing a new process on a pilot scale for "Microquartz" production. Process changes are expected to produce stronger, purer fibers, as follows:

1. Lead leaching tanks have been replaced with stainless steel tanks. (Sulfuric acid is still used for leaching because of low volatility.)

2. Fibers will probably be dewatered by centrifugation rather than roll-pressing. This change should result in less fiber breakage and could result in increased filter strength.

3. Gas-fired conveyor dryer has been replaced with an electrically heated tumbling dryer. This change is intended to increase fiber purity.

Toward the end of this program, we learned that ICI has developed high-purity alumina and zirconia fibers similar in size and price to "Micro-quartz," but we have not evaluated them. Another candidate fiber is being developed by 3M Co. Production quantities of the ICI fibers are scheduled to be av.ilable in the latter half of 1974.

C. ATOMIC ABSORPTION AND FLAME EMISSION SPECTROMETRY

Atomic absorption and flame emission are but two members of a broad class of techniques in which the sample is first vaporized to produce dissociated atomic species. The atoms are then electronically excited, and either the absorption or emission of radiation (in the visible or ultraviolet region) associated with the electronic transition is measured and related to concentration.

In flame emission, the functions of vaporization, dissociation, and electronic excitation are all performed by the flame itself. As the electronically excited atoms relax to their ground state, they emit radiation of a wavelength characteristic of the particular atom and transition. In atomic absorption, in its most commonly employed form, the function of the flame is only to vaporize the sample and produce ground state atomic species. The electronic excitation is produced by a source of monochromatic light, usually a hollow cathode discharge tube; the amount of light absorbed is measured and related to the atomic concentration.

For the alkali and alkaline earth metals, flame emission is the more sensitive of the two techniques; for other species, the reverse is usually true. The relative sensitivities of the two techniques depend largely on the ionization potential of the element in question. Alkali metals have a low ionization potential and are easily excited, even in relatively low temperature air/acetylene flames. As one moves to the right across the periodic table, ionization potentials increase, the flame becomes less effective in producing ionization, and sensitivity falls. For elements with higher ionization potentials, some flame emission sensitivity can be regained by using a hotter flame such as nitrous oxide/acetylene. In atomic absorption, electronic excitation by the flame is disadvantageous, since a large proportion of the atoms must be initially in their ground states if a significant amount of the exciting hollow-cathode radiation is to be absorbed and consequently measured.

Both techniques, as commonly practiced, involve the aspiration into the flame of a solution of the sample to be measured. However, there are a variety of approaches by which atomic absorption measurements may be made without utilizing a flame. Probably the best known is the "cold vapor" technique in which mercuric ion in solution is first reduced to metallic mercury by stannous ion, and the metallic mercury is then sparged out of the solution with air or nitrogen. The gas stream containing the mercury vapors is passed into a gas cell, and the ultraviolet absorption is measured. Elimination of the flame and its associated noise permits one to achieve a mercury detection limit of a few nanograms. Other alternatives to flame vaporization include electrically heated filaments and graphite furnaces.

D. X-RAY FLUORESCENCE ANALYSIS

When a sample is irradiated by sufficiently energetic photons, usually X rays from a tube with a chromium or tungsten target inner-shell electrons of the sample atoms are completely ejected from the atom. The resulting high-energy vacancy is then immediately filled by an electron from an outer shell of the atom, and a secondary X-ray photon is emitted. The energy of this "fluorescent" X ray is proportional to the atomic number of the element and also depends upon the particular valance shell which is filled.

The sensitivity of analyses by X-ray fluorescence depends both on the efficiency with which the exciting X rays are absorbed and also on the efficiency with which the fluorescent X-radiation can escape to the detector. Elements with an atomic number less than 11 (sodium) are quite transparent to the exciting X rays and, hence, cannot be measured.

X-ray fluorescence, like neutron activation discussed in the following section, is usually employed as a nondestructive analytical tool in which the entire sample, filter plus collected particulate, is subjected to analysis. Thus, in contrast to atomic absorption and flame emission, the total level of impurities in the bulk filter must be considered.

X-ray fluorescence measurements are extremely sensitive to the presence of other elements in the sample matrix. The presence of small amounts of a heavy metal is of particular concern when one is attempting to measure a lighter species. Matrix effects also become very important when one is trying to measure a sample thicker than 10 to 100 microns, as is the case in the direct measurement of particulate trapped throughout deep filters (particularly if materials such as glass, which itself absorbs X-radiation, is used in constructing the filter).

E. NEUTRON ACTIVATION ANALYSIS

Radioactivation consists of the absorption of an elementary particle, such as a neutron, a proton, or an alpha-particle, into the nucleus of an atom; the latter becomes unstable and spontaneously emits electromagnetic radiation and/or energetic particles. By observing the energies and intensities of emission, both qualitative and quantitative analytical information can be obtained. Neutron activation is the most common and most generally useful. This reaction converts atoms of a particular isotope to the next higher isotope of the same element. Thus, for example, sodium-23 absorbs thermal neutrons to become radioactive sodium-24. The subsequent radioactive decay, involving emission of both beta and gamma rays, yields stable ^{24}Mg.

For analysis of particulates collected on porous filters, the neutron-activation method can be applied to samples "as received," provided that the filter substrate is sufficiently free of the elements sought, and that it is suitable for exposure to an intense neutron flux. Filters made only of hydrocarbons, such as polyethylene or polypropylene, are

ideal because of the complete insensitivity of carbon and hydrogen to neutron bombardment. Filters containing nitrogen and oxygen are satisfactory because these elements are insensitive to thermal neutrons.

Silica fiber filters are susceptible to neutron activation and require more careful consideration. One isotope, ^{30}Si, will produce ^{31}Si, with a half-life of 2.6 hours; its radiation could be troublesome in the case of long irradiations of several days or weeks. The decay of ^{31}Si is almost entirely by beta radiation, which is easily distinguished from the gamma radiation of analytical value.

When long irradiations are needed to activate long-lived elements in the particulate samples (Fe, Cr, Zn, etc.), any interference produced by the ^{31}Si radioactivity that cannot be suppressed by the detector itself can be eliminated by suitable radiochemical separation. Radiochemical separation for other long-lived nuclides may be equally advantageous.

Facilities for conducting neutron-activation analysis should be capable of producing a thermal neutron flux of at least $10^{12} n/cm^2$ sec in order to achieve adequate sensitivity for all important trace-elements. Availability of comparable fast-neutron fluxes of known intensity (separate from the thermal-neutrons) is also desirable for some special situations. Ideally, the analysis would be done without chemical separation and would, therefore, require the availability of a high-resolution (Ge:Li) semiconductor gamma spectrometer facility, equipped with special computation capabilities for direct calculation of trace-element concentrations and correction of matrix-interference effects wherever they may occur.

F. EXTRACTION OF FILTERS FOR ANALYSIS

For atomic absorption and flame emission analyses, particulates on filters must first be put into solution by simple dissolution in water, acid digestion, fusion, or leaching with solutions containing complexants. The fact that a dissolution step is required can ease the requirements for filter purity in two ways. First, only a small proportion of certain of the impurities within the filter may actually leach out during dissolution of the sample. Secondly, impurity levels in the reagents required for dissolution may become limiting factors before impurities leaching out of the filter itself become important.

Filter extraction should be either preceded by or combined with an ashing step to remove organic matter. Three types of ashing procedures currently used for analysis of particulates on filters are as follows:

1. Ashing in a Muffle Furnace at 500°C

Past studies have indicated serious losses of certain metals, namely, lead, zinc, cadmium, and copper. The cause of these losses has been ascribed to either vaporization or formation of acid-insoluble silicates. A recent study[7] provides good evidence for the latter and indicates that muffle furnace ignition in the absence of silicates (i.e., paper filters

in platinum ware) gives good recovery of the above metals.

2. Low-Temperature Plasma Ashing at 50 to 250°C in a Low-Pressure Oxygen Atmosphere

Thompson et al[8] show data which indicate that plasma ashing minimizes losses of most metals, but there is still some question regarding losses of highly volatile species such as arsenic and mercury.

3. Wet Ashing with Nitric-Perchloric Acid Mixtures

Wet ashing combines the ashing and extraction steps and gives similar results to plasma ashing.[7] However, the acids are somewhat hazardous and must be of very high purity in order to avoid blank problems.

As a filter purification step, any of these or other ashing procedures can be used. For example, ashing may be combined with heat treatment to increase purity and strength.

Ideally, a universal solvent for the particulates of interest, which does not dissolve silica, should be used both for filter purification and particulate analysis. In practice, however, a variety of solvents will probably be used, depending on the particulate elements to be analyzed. We have therefore reviewed the analytical methods for particulates containing metallic compounds to guide the selection of a filter purification and analytical extraction procedure.

Procedures used to dissolve particulates have generally involved treatment of the previously ashed sample for 1 to 3 hours with hot (redistilled) nitric and/or hydrochloric acids. The most frequently used reagents are nitric acid (30% to 60% concentrations) and a nitric hydrochloric acid mixture (32 ml of 40% HNO_3 + 8 ml of 19% HCl). In one study, dustfall was treated with nitric, hydrochloric, and perchloric acids and mixtures of these acids, and hydrochloric was the most successful for extracting Fe, Cu, Pb, and Zn.[9] However, the complete solubility of oxides of elements such as aluminum, boron, and zirconium in any of these acids is questionable.

A possible alternative extractant of analytical interest is hydrofluoric acid, which would dissolve virtually all of the elements of interest. However, use of HF presents other problems, including the need for platinum containers, the complete dissolution of the glass filter (which will release all impurities contained in the glass fiber), and the possible loss of some metals by volatilization.

Perchloric acid offers no obvious advantages over nitric acid in extraction mixtures but would likely be included in any wet ashing procedure. The presence of at least some hydrochloric acid may be useful since this will function as a complexant for many of the metal species, and may aid in stabilizing the very low concentrations expected.

Based on the above review, we decided to use a 1- to 3-hour boiling, high-purity, nitric-hydrochloric acid treatment for analytical extraction. This treatment will dissolve the majority of possible compounds of the metals of interest.

G. MINIMUM DETECTABLE CONCENTRATIONS

Detection sensitivities for most elements of concern when measured by atomic absorption, flame emission, and X-ray fluorescence spectrometry as well as neutron activation analysis are presented in Table 1.

A comparison of the detectabilities shown in Table 1 indicates that atomic absorption is probably a more important technique for trace analysis of air pollutant particulates than flame emission. In only a few instances (the alkali metals and alkaline earths) is flame emission more sensitive than atomic absorption.

It must be stressed that the levels presented in Table 1 are generally the lowest attainable detection limits. The presence of one or more of a wide variety of chemical interferences and/or imperfect optimization of instrument operating parameters can, and usually do, markedly decrease the sensitivity which is actually obtained. For practical purposes, the detection limits normally obtained in most laboratories are probably up to 10 times higher than the levels presented in the table.

In order to establish filter purity goals, we computed the maximum concentrations of elements which should be allowed in the filter based on the best atomic absorption and flame emission detection limits, normal extraction procedures, and the normal filter weight. This list, presented in Table 2, gives the minimum concentration which can be detected by atomic absorption and flame emission in 25 ml of extract from a single 4-inch diameter (0.85 g) filter.

TABLE 1

COMPARATIVE MAXIMUM SENSITIVITIES
FOR ATOMIC MEASUREMENT TECHNIQUES

Element	Detection Limit[a]			
	AAF μg/ml[b]	FES μg/ml[c]	XRF μg/cm²[d]	NAA μg[e]
Mg	0.003	0.005	0.1	0.5
Al	0.04	0.005	0.1	0.004
K	0.005	0.0005	0.1	0.2
Ca	0.0005	0.0001	0.01	4
Ti	0.1	0.2	0.01	0.1
V	0.02	0.01	0.01	0.002
Cr	0.005	0.005	0.01	0.3
Mn	0.002	0.005	0.01	0.0001
Fe	0.005	0.05	0.01	2
Co	0.005	0.05	0.01	0.01
Ni	0.005	0.03	0.01	0.7
Cu	0.005	0.01	0.01	0.002
Zn	0.002	0.002	0.01	0.1
As	0.1	50	0.01	0.005
Zr	5	3	0.01	0.8
Mo	0.03	0.1	0.01	0.1
Ag	0.005	0.02	0.1	0.004
Cd	0.005	2	0.1	0.005
In	0.05	0.002	0.1	0.00006
Sn	0.06	0.5	0.1	0.03
Sb	0.1	20	0.1	0.007
Te	0.3	200	0.1	0.03
Ba	0.05	0.001	0.1	0.02
Hf	NA	NA	0.1	0.0006
Ta	5	5	0.1	0.1
W	3	0.5	0.1	0.004
Hg	0.2	40	0.1	0.08
Pb	0.03	0.2	0.1	0.5

NA - Sensitivity not available.

(a) Under optimum conditions, presence of interferences or use of other than optimum instrumental operating parameters can produce significant individual or overall degradations (see text).

(b) Atomic absorption with flame (Source, Refs. 10, 11).

(c) Flame emission (Source, Refs. 10, 11).

(d) X-ray fluorescence (Source, Ref. 12).

(e) Neutron activation, weight of total sample can be in range of 0.1-10g (Source, Ref. 13).

TABLE 2

MINIMUM DETECTABLE CONCENTRATIONS OF IMPURITIES IN GLASS FIBER FILTERS*

	Atomic Absorption			Flame Emission		
Element	µg in Extract	µg per g of Fiber	µg per sq. ft.	µg in Extract	µg per g of Fiber	µg per sq. ft.
Ag	0.13	0.16	1.6	0.5	0.6	6
Al	1.0	1.2	12	0.13	0.16	1.6
As	2.5	3	30	1250	1500	15000
B	1000**	1200	12000	NA		
Ba	1.3	1.6	16	0.025	0.03	0.3
Be	0.75**	0.9	9	NA		
Ca	0.013	0.016	0.2	0.0025	0.003	0.03
Cd	0.13	0.16	1.6	50	60	600
Co	0.13	0.16	1.6	1.3	1.6	16
Cr	0.13	0.16	1.6	0.13	0.16	1.6
Cu	0.13	0.16	1.6	0.25	0.3	3
Fe	0.13	0.16	1.6	1.3	1.6	16
Hf	380**	450	4500	NA		
Hg	0.03**	0.04	0.4	1000	1200	12000
In	1.3	1.6	16	0.05	0.06	0.6
K	0.13	0.16	1.6	0.013	0.016	0.2
Mg	0.075	0.09	0.9	0.13	0.16	1.6
Mn	0.05	0.06	0.6	0.13	0.16	1.6
Mo	0.75	0.90	9	2.5	3.0	30
Ni	0.13	0.16	1.6	0.75	0.9	9
P	6300**	7500	75000	NA		
Pb	0.75	0.9	9	5.0	6.0	60
Rh	0.75	0.9	9	0.5	0.6	6
Sb	2.5	3.0	30	500	600	6000
Sn	1.5	1.8	18	13	16	160
Ta	125	150	1500	125	150	1500
Te	7.5	9.0	90	5000	6000	60000
Ti	2.5	3.0	30	5.0	6.0	60
V	0.5	0.6	6	0.25	0.3	3
W	75	90	900	13	16	160
Zn	0.05	0.06	0.6	0.05	0.06	0.6
Zr	125	150	1500	75	90	900
Na	0.05	0.06	0.6	0.01	0.012	0.1
Si	2.5	3	30	125	150	1500

NA = Non-applicable.

*Detection limits based on extraction of a 4-in. diam filter weighing 0.85 g (density = 10 g per sq ft), a final extract volume of 25 ml, and detection limit data given in Table 1.

**Detection limits as reported in Atomic Absorption Methods Manual, 1971, Perkin-Elmer Corporation.

III. ANALYSIS AND PURIFICATION OF SILICA FIBERS

A. CHEMICAL ANALYSIS APPROACH

Since the detection limits of each of the analytical techniques of interest is known, it was not necessary to analyze the filters by each of these techniques all of the time. The need was simply to show that the elements of interest are present below the detection limits. Initially, our objectives were:

1. To survey candidate fibers for acid-extractable impurities for comparison with AAS and FES detection limits

2. To observe the effects of possible processing steps (e.g., leaching and ignition) on the amounts of acid-extractables.

Because of the large number of elements to be considered (32) and the high cost of complete AAS analyses, we used emission spectrography to estimate the impurities in portions of the dried acid extracts, and used these data to select a few key elements for further analysis by AAS.

When final candidate filters were prepared, they were extracted and analyzed for key elements via atomic absorption spectrometry. They were also analyzed by X-ray fluorescence and neutron activation in a preliminary way.

B. EXTRACTABLE IMPURITY SURVEY BY EMISSION SPECTROGRAPHY

In order to estimate purity of some candidate fibers and to test the effect of acid leaching, we carried out a series of three extractions and emission spectrographic analyses on each of the following types of high-purity quartz fibers:

1. Code 108 Johns-Manville 99.2% silica "Microquartz" (0.7 to 1.6 μm diameter fibers of unspecified length)

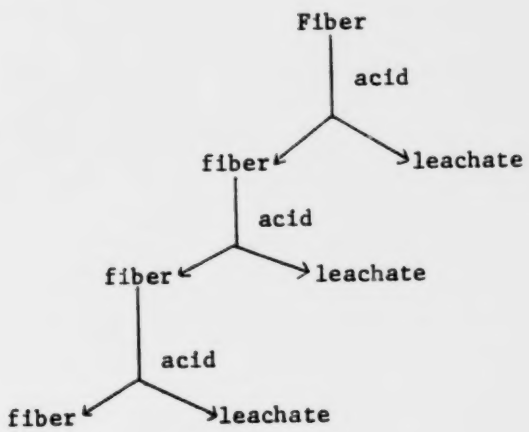
2. General Electric quartz (10 μm diameter continuous filament)

3. J. P. Stevens Standard Astroquartz Mat (7 to 9 μm diameter crimped fibers bonded with 4 to 5% polyvinyl alcohol). Purity was greater than 99.95%.

The fibers were extracted without prior ashing except that a duplicate portion of the GE fiber was heated at 500°C for one hour and then carried along in the extraction scheme. The leachings were performed in both Vycor and Pyrex containers (50 and 100 ml crucibles and beakers).

Separate blanks were carried through for both Vycor and Pyrex vessels. Prior to use, all glassware and equipment were soaked in 6N HCl, and then rinsed well with distilled water.

The extraction sequence was:



The acid solvent was a dilute aqua regia prepared by mixing 45 ml of 70% HNO_3, 10 ml of 37% HCl, and 45 ml of deionized water. The acids were ACS Reagent Grade (Fisher Scientific Co.).

The samples were transferred into the extraction vessels with a minimum of handling using cleaned glass and polyethylene tools. The continuous filament GE fiber was cut with a pair of stainless steel surgical scissors after winding an appropriate amount onto a polyethylene rod. Weighed amounts of fiber were used which could be fully covered by 30 ml of extraction solvent.

The extraction vessels were covered with watchglasses and heated on a hot plate at just below the boil for one hour. The hot plate was set in a large hood and was protected on top and in front by sheets of polyethylene mounted on a frame to minimize "fallout" into the samples.

The extract was transferred with the aid of a clean glass rod into a

clean container for evaporation and the extraction was repeated with a fresh 30 ml portion of solvent. This second extract was transferred into another clean container and a third extraction carried out. Following the third extraction and transfer, the fiber mass was washed well with deionized water and dried in a clean container on the hot plate.

All extracts were evaporated to 5 ml volume, using a hot plate at a rate to avoid spattering. Then approximately 25 mg of lithium nitrate matrix agent (Johnson Matthey, spectrographically analyzed) were added to each and evaporation to dryness was completed. The lithium salt was added to facilitate emission spectrographic estimation of alkali and alkaline earth elements and to aid the mechanical collection of the residue.

The residues were scraped up and removed with cleaned polyethylene tools to avoid any abrasion of the glass, and submitted to Kent Laboratories for emission spectrographic analysis. In addition, portions of the neat and extracted fibers and a full set of reagent and container blanks were submitted. Results are listed in Tables 3 and 4. The circled values in Table 3 are near or below the AAS detection limits.

With few exceptions, the amounts of impurities in Table 3 vary little from the first to the third extract, indicating that the impurities are likely distributed within the fiber, rather than present as an easily removed surface contamination.

The fiber analyses (Table 4) indicate no great decrease in impurity levels resulting from acid extractions. This also suggests that the impurities are distributed uniformly in the fiber and difficult to extract. The apparent large increase in sodium after acid extraction is an anomaly, but sodium is not one of the trace elements under consideration.

C. EFFECT OF HEATING ON EXTRACTABLE IMPURITIES

Table 3 also suggests that heating the fibers to 500°C reduces the extractable impurity content. We therefore used atomic absorption spectrometry (AAS) to verify the preliminary emission spectrography results. Fibers used in these experiments were:

1. 99.2% silica Code 106 Johns-Manville "Microquartz." This fiber was not previously tested. It is the smallest diameter silica fiber of this purity available from Johns-Manville.

2. 99.2% silica Code 108 Johns-Manville "Microquartz" (same lot used in previous tests).

3. 99.98% silica J. P. Stevens Chopped Astroquartz. This fiber is 1/4" long and was not previously tested. The previously tested J. P. Stevens Astroquartz was a different lot of less certain purity (over 99.95%), and was in mat form with 4-5% polyvinyl alcohol binder.

TABLE 3

EMISSION SPECTROGRAPHIC ANALYSES OF ACID EXTRACTS FROM FIBERS

Element	AA Detection Limit* µg per g of fiber	Emission Spec. Detection Limit-µg	Impurities Found in Successive Acid Extracts - µg per g of fiber												
			J.P.S. Astroquartz (Sample wt. = 0.2g) Extraction				J-M Microquartz Code 108 (Sample wt. = 1.0g) Extraction				GE Quartz Fiber (Sample wt. = 0.25g) Extraction				GE Quartz Fiber Heated 1 hr. @ 500°C Extraction
			First	Second	Third	Total	First	Second	Third	Total	First	Second	Third	Total	Total**
Aluminum	1.2	0.2	45	70	15	130	26	28	3	57	2.5	30	ND	33	13
Barium	1.6	0.05	ND	ND	ND	-	1	0.07	ND	(1)	ND	ND	ND	-	ND
Boron	1200	0.5	0.8	0.5	ND	(1)	35	8	0.7	(45)	0.5	0.6	ND	(1)	ND
Calcium	0.02	0.1	17	8.5	34	60	0.6	2	1	3.6	8	4	0.4	12	10
Chromium	0.2	0.05	4.5	7.5	2.5	15	ND	0.3	ND	(0.3)	4.4	3.2	0.8	8.4	ND
Copper	0.2	0.05	7	3	65	75	2.8	0.5	1.2	4.5	12	ND	ND	12	23
Iron	0.2	0.1	29	18	17	64	2.4	3.9	2.3	8.6	14	12	10	36	31
Lead	0.9	0.1	0.7	ND	ND	(0.7)	11	7	6	24	4	2	0.4	6.4	ND
Magnesium	0.09	0.1	17	4.5	3	25	0.8	1.2	0.7	2.7	6.4	0.8	ND	7	ND
Manganese	0.06	0.01	ND	ND	ND	-	0.15	0.08	0.03	0.26	0.34	0.16	0.06	0.56	ND
Nickel	0.2	0.1	0.9	0.5	0.1	1.5	ND	0.1	ND	(0.1)	0.2	2.4	1	3.6	ND
Silver	0.2	0.01	2	1	0.7	3.7	ND	ND	0.3	(0.3)	0.5	0.2	0.4	1.1	7.5
Tin	2	0.1	7	1.2	2.5	11	1.0	0.4	15	16	6.4	ND	ND	6.4	ND
Titanium	3	0.1	5	5	10	20	0.2	0.3	1.1	(1.6)	3	3	ND	(6)	(2.6)
Zinc	0.06	2	ND	ND	ND	-	ND	ND	ND	-	ND	ND	ND	-	ND
Zirconium	150	0.5	ND	ND	ND	-	ND	ND	ND	-	ND	ND	ND	-	ND

ND = Not detected.

◯ = near or below AA extraction detection limit.

*From Table 2.

**Total impurities found in first, second, and third extracts of 0.2 g fiber.

TABLE 4

EMISSION SPECTROGRAPHIC ANALYSES OF QUARTZ FIBERS
BEFORE AND AFTER ACID EXTRACTION
(μg per g of fiber)

Element	J.P.S. Astroquartz		J-M Microquartz		GE 10μ Fibers		Heated GE Fibers*
	Before	After	Before	After	Before	After	After Extraction
Sodium	<1	5000	300	1000	<1	2000	3000
Aluminum	10	10	1000	1000	6	10	10
Lead	<0.5	<0.5	300	1000	<0.5	<0.5	<0.5
Iron	10	100	200	200	10	10	20
Titanium	<0.5	<0.5	100	100	<0.5	<0.5	<0.5
Calcium	10	30	70	70	10	8	10
Magnesium	3	1	30	30	3	1	1
Nickel	3	20	20	1	3	1	3
Zirconium	<0.5	<0.5	20	20	<0.5	<0.5	<0.5
Barium	1	<0.1	10	10	0.5	<0.1	<0.1
Strontium	<0.1	<0.1	10	10	<0.1	<0.1	<0.1
Chromium	<0.1	<0.1	5	5	<0.1	3	<0.1
Copper	3	3	3	3	3	1	2
Silver	0.3	1	1	<0.1	0.5	0.5	0.5
Zinc	20	<1	<1	<1	<1	<1	<1
Manganese	<0.1	<0.1	1	1	<0.1	<0.1	<0.1

*Sample ignited at 500°C for 1 hour before extraction.

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4. General Electric 10 μm Quartz Fiber (same lot used in previous test).

Samples of 2.0 g ± 0.01 g each of the fibers were ignited for one hour at either 540°C or 800°C in previously ignited Vycor crucibles. The fibers were then extracted, as previously described, with 60 ml of dilute aqua regia for three hours at just below the boil. The extract was then decanted off, evaporated nearly to dryness to eliminate much of the acid and diluted to 50 ml with deionized water. Some samples were extracted again for one hour in the same manner.

The extracts were quantitatively analyzed by AAS for lead, zinc, copper, aluminum, and iron. The results are shown in Tables 5 and 6. These results were corrected for blank analyses (no fiber). Pyrex and Vycor beaker blanks were almost identical; therefore, we used acid-washed Pyrex beakers for most of these analyses.

During these analyses, filter handsheet development work progressed to the point where the Johns-Manville "Microquartz" was identified as the best available fiber for making filters. Therefore, whereas analytical results on all fibers are reported, we will emphasize the "Microquartz" results in interpreting the data.

The Code 106 "Microquartz" appears to be slightly purer than the Code 108.

Heating the fibers to 540°C did not reduce the extractable metal contents but heating to 800°C looks promising, as follows:

1. Extractable zinc and copper levels in J-M "Microquartz" are reduced to our AAS detection limit by this treatment.

2. Extractable lead levels in J-M "Microquartz" are reduced by 1/2 or 2/3 by one hour at 800°C, but are still above the minimum detection limit.

Extractable iron levels in J-M "Microquartz" are not reduced by preheating. Extractable aluminum levels in J-M "Microquartz" were at the detection limit before heating but doubled after preheating. The increase may be due to sample contamination with alumina from the furnace lining.

It should be noted that the practical detection limits (signal/noise ratio ≈2) of our AAS equipment (Perkin-Elmer Model 303 with Recorder Readout Accessory and Hewlett-Packard Model 7127A Recorder), as shown in Tables 5 and 6, are about three to four times higher than those given in Table 2. However, these detection limits are probably characteristic of the real day-to-day variation in limits which can be attained in a working laboratory with common types of currently used equipment.

To determine the effect of heating the fibers to 800°C on the other extractable elements, a complete qualitative metals survey was done by emission spectrographic analysis. These samples were prepared by

TABLE 5

EFFECT OF HEATING FOR 1 HOUR ON EXTRACTABLE IMPURITIES IN QUARTZ FIBERS

	Impurities Measured by AAS - μg per g of Fiber								
	Lead			Zinc			Copper		
	Not Ignited	550°C	800°C	Not Ignited	550°C	800°C	Not Ignited	550°C	800°C
J-M Microquartz Code 106	19	18	<12* 5.5	0.45	0.25	<0.25 <0.25	0.8	<0.8	<0.8 <0.8
J-M Microquartz Code 108	--	33	18	--	0.5	<0.25	--	<0.8	<0.8
J.P.S. Astroquartz	<2.5	<12*	<2.5	0.35	0.5	0.25	0.8	<0.8	<0.8
GE 10 micron fiber	<2.5	<12*	<2.5	1.1	1.8	0.37	1.6	2.5	<0.8

*Detection limit for older lead hollow-cathode lamp replaced part way through tests.

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TABLE 6

EFFECT OF HEATING ON EXTRACTABLE IMPURITY LEVELS OF CODE 108 MICROQUARTZ[a]

Element	AAS Detection Limit µg per g	Impurities Measured by AAS - µg per g of Fiber			
		Unheated Fiber		After 1 hr @ 800°C	
		1st Extraction	2nd Extraction	1st Extraction	2nd Extraction
Lead	4[b]	39	15	13	<4
		38	12	11	7
Iron	0.4	7.1	6.0	15	9
		11	6	8	6
Aluminum	11	<11	<11	22	26
		13	19	23	13

a. Corrected for reagent blanks.

b. The difference between this value and the 2.5 value in Table 5 reflects daily variation in detection limit.

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evaporating 25 ml aliquots of the same diluted extracts that were used in Table 5, adding 20 mg of high-purity lithium nitrate as a carrier, and evaporating to dryness. Results are shown in Table 7. They have been approximately corrected for the "blank" by eliminating those elements which were found to be present either at the same or at a higher level as a reagent blank (extraction acids plus lithium nitrate carrier) carried through the same procedure. The qualitative results on the acid extracts have been related approximately to the level of extractables in the original fiber sample by multiplying the extract solids level by 0.02 (extract from 1 gram of fiber was mixed with 0.02 grams of lithium nitrate carrier prior to analysis).

Table 7 indicates that the only "Microquartz" impurities exceeding the AA detection limits in Table 2 are aluminum, iron, titanium, and chromium. However, AA data in Tables 5 and 6 indicate that extractable lead levels also exceed AA detection limits.

Table 8 shows an overall comparison of acid-extractable materials in the three candidate fibers before and after heating. The data for the non-ignited fibers is taken from Table 3. These data indicate that the levels of acid-extractable species are generally one order of magnitude lower after ignition to 800°C for one hour. The decrease agrees with the decrease shown by quantitative atomic absorption measurements of lead, zinc, and copper in the same extracts reported in Tables 5 and 6.

Table 8 agrees with previous AA results on iron which indicate no reduction due to heating, and the tenfold reduction in "Microquartz" aluminum level shown in Table 8 suggests that the doubled aluminum concentration indicated by AA in Table 6 was due to contamination.

This section indicates that 99.2% Johns-Manville "Microquartz" meets almost all extractable impurity requirements after heat treatment for one hour at 800°C.

D. EFFECT OF MACHINE MANUFACTURING PROCESS ON EXTRACTABLE FILTER IMPURITIES

Up to this point, chemical analyses on this program have been limited to candidate fibers for use in the filter. As a guide to planning the analysis of several filter handsheets by atomic absorption, we have attempted to estimate the kinds and amounts of acid-extractable impurities which could be added to a filter during machine manufacturing by analyzing both the fibers and the finished filters.

We know that Pallflex "Tissuquartz" is made by machine. We understand that the machine is almost entire stainless steel and that "Tissuquartz" consists primarily of 98.5% SiO_2 Johns-Manville Code 106 "Microquartz." Ideally, we should have analyzed the same lot of raw material (fiber) and finished product (filter) to determine the effect of manufacturing on extractable impurities. However, the best raw material samples we could obtain were from Johns-Manville Co. and in larger sizes (98.5% Code 108

TABLE 7

QUALITATIVE COMPOSITION OF ACID EXTRACTS FROM FIBERS HEATED AT 800°C

Approximate Level in Extract Solids[b]	Approximate Level in Fiber[c]	Code 108 J-M Microquartz[a]	J.P.S. Astroquartz[a]	GE Quartz Fiber[a]	Elements in Reagent Blank
>10%		Si	Si		Li
0.1-1%				Si	
100-1000 ppm	2-20 ppm	Al, Fe			
30-300 ppm	1-10 ppm	Ti	Al, Fe, Ti	Fe	
10-100 ppm	0.2-2 ppm	B, Cr			Fe, Na
3-30 ppm	0.1-1 ppm	Pb	B, Cr	Al, Cr	Ca, Cu, Mg
1-10 ppm	0.02-0.2 ppm		Pb	B, Pb	Al, Cr, Ni Si, Ti
0.3-3 ppm	0.01-0.1 ppm	Ba, Mn	Ba, Mn	Mn	Pb

[a] Corrected for reagent blank (acids plus lithium nitrate).

[b] This is estimate of level in extract solids which are primarily lithium nitrate.

[c] Calculated by multiplying level in extract solids x $\frac{0.02 \text{ (wt of solids)}}{1 \text{ (wt of fiber)}}$.

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TABLE 8

QUALITATIVE COMPOSITION OF ACID EXTRACTS FROM AS IS AND IGNITED[(a)] FIBERS

Approximate Level of Acid Extractables in Fiber	J-M Microquartz		J.P.S. Astroquartz		GE Quartz	
	As is	Ignited[(a)]	As is	Ignited[(a)]	As is	Ignited[(a)]
>100 µg/g			Al			
10-100	Al, Pb, B		Cr, Cu, Fe Ca, Mg		Al, Cu, Fe Ca	
1-10	Cu, Fe, Sn Ti, Ca, Mg	Al, Fe, Ti	Ni, Ag, Sn Ti	Al, Fe, Ti	Cr, Pb, Ni Sn, Ti, Mg	Fe
0.1-1	Cr, Mn, Ni Ag, Ba	Cr, Pb, Cu B, Ca, Mg	Pb, B	Cr, Cu, B Ca, Mg	Mn, Ag, B	Al, Cr, Cu Ca, Mg
.01-0.1		Mn, Ni, Ba	Mn, Ba	Pb, Mn, Ni Ba	Ba	Pb, Mn, Ni Ti, B
>.01						

(a) Ignited for 1 hour at 800°C.
Sn not detected (limit ca. 1 µg per g).
Ag not detected (limit ca. 0.01 µg per g).

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and Code 112 "Microquartz").

We analyzed the "Microquartz" fibers and "Tissuquartz" filters by the emission spectrography procedure reported in Section III.B. Samples of 1.0 g each were extracted for three hours with 60 ml of hot dilute aqua regia. The extract was decanted off and evaporated to about 2 ml. Then 0.020 g of high-purity lithium nitrate was added and the remaining liquid was evaporated. The solids were then analyzed by Kent Laboratories. Results are presented in Table 9. The data have been corrected for elements found in a reagent blank carried along with the samples.

The data in Table 9 indicate that the Pallflex manufacturing process has very little effect on acid-extractable impurities. The major differences in impurity levels are a slight increase in tin in "Tissuquartz" Roll No. 2 compared to both "Microquartz" fibers, and a slight decrease in lead in both "Tissuquartz" rolls compared to Code 108 fiber. However, because the fibers and filters were not actual raw material and finished product samples, these differences are not considered significant. We conclude only that the Pallflex filter manufacturing process probably does not greatly affect the purity of the raw material fibers.

TABLE 9

QUALITATIVE COMPOSITION OF ACID EXTRACTS FROM 98.5% SiO_2 FIBERS AND FILTERS

Approximate Level in Extract Solids[2]	Approximate Level in Fiber[3]	Code 108 J-M "Microquartz"[1]	Code 112 J-M "Microquartz"[1]	Roll #2 Pallflex[1]	Roll #3 Pallflex[1]	Elements in Reagent Blank
>10%						Li
1-10%			Si		Si	
0.3-3%	0.01-0.1%	Si, Na	Na	Si, Na	Na	
0.1-1%	20-200 ppm	K				
300-3000 ppm	10-100 ppm	Ba, Pb		K, Sn	K	Na
100-1000 ppm	2-20 ppm	Ca, Cu	Sb, Ca, Mg	Ca, Mg	Ca, Mg	K, Fe
30-300 ppm	1-10 ppm		Pb	Cu, Pb	Sb, Cu, Pb	Ca
10-100 ppm	0.2-2 ppm	Sn, Ti	Ba, Sn, Ti, Ni	Ni, Ti	Ba, Cr, Sn, Ni	Cu, Mg, Si, Pb
3-30 ppm	0.1-1 ppm	Ni	Al, B		Ti	Ba, Cr, Sn, Ni
1-10 ppm	0.02-0.2 ppm				B	Mn

[1] Corrected for reagent blank (acids plus lithium nitrate)

[2] Estimate of concentration level in extract solids which are primarily lithium nitrate

[3] Calculated by multiplying level in extract solids x $\frac{0.02 \text{ (wt of lithium nitrate solids)}}{1 \quad \text{(wt of fiber taken)}}$

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IV. FILTER HANDSHEET DEVELOPMENT

As indicated in Section II.B, high-efficiency quartz fiber filters are commercially available but are too weak and moisture-sensitive for gravimetric measurements. We view the low strength (about 0.4 lb/in.) as the principal problem and attribute it to the use of fragile, porous (leached glass) fibers, the absence of a binder, and weak fiber-to-fiber attractive forces. The filter can be strengthened by heat treatment at about 1000°C but it becomes brittle and non-foldable. We therefore focused our efforts on increasing strength without loss of purity or flexibility.

Filter handsheets were made by dispersing fibers in water, pouring the slurry into a papermaking mold, and draining the water (through a woven wire screen) to form a fibrous web. Strength was measured with an Instron Tester at a jaw gap of 2" and a crosshead speed of 2"/min. We measured filter efficiency and flow resistance with the liquid dioctyl phthalate (DOP) smoke test (ASTM D 2986-71).

A. FILTER STRENGTH VS. FIBER BEATING CONDITIONS, HEATING TIME, AND FIBER LENGTH

A standard laboratory method of preparing aqueous fiber slurries in filter development work is to use a Waring blender. We expected this method to produce severe fiber damage, so we investigated several beating conditions, as shown in Table 10.

In these experiments, 0.8 g of Code 106 and 1.2 g of Code 108 Johns Manville 99.2% silica "Microquartz" fibers were added to 2000 ml of distilled water in a gallon-size stainless steel Waring blender and adjusted to pH 2-3 with 1:1 HCl. After beating, the slurries were cast into a 6" diameter handsheet mold, gravity-drained, vacuum damp-dried, and dried between blotters at 116°C on a drum drier. The results suggested that 3-5 minutes beating at low speed produced stronger filters than either less severe or more severe beating.

As previously discussed, chemical analysis and purification studies indicated that heat treatment at 800°C for 1 hour greatly reduced extractable impurity levels of many metals. We therefore prepared samples as above and determined the effect of 800°C heat treatment on filter strength. The results, as shown in Table 11, indicate that 30 minutes was optimum but inadequate (0.24 lbs/in. tensile strength).

The silica fibers used above are air blown and therefore of variable unknown lengths. The average length, however, is believed to be well under 1/4". Because fibers of 1/4" length or greater generally increase filter strength, we prepared additional samples as above, except that we replaced half of the Code 108 fibers with 1/4"-long, 7-9 μm diameter Astroquartz fibers (40% Code 106, 30% Code 108, and 30% Astroquartz). Both as-received Astroquartz (silane-coated) and heat-cleaned Astroquartz

TABLE 10

EFFECT OF FIBER DISPERSION CONDITIONS
ON FILTER STRENGTH

Filter Sample	Speed	Voltage	Time	Tensile Strength lb/in.	Fiber Loss* wt %
17-A	High	110	3	0.17	13.5
15-B	Low	110	1	0.13	7.5
17-B	Low	110	3	0.17	13.5
21-A-1	Low	110	5	0.19	14.0
21-A-2	Low	110	10	0.10	23.5
17-C	Low	60	1	0.13	7.5
17-D	Low	40	1	<<0.10**	--

*Wt % of fibers lost through the screen during handsheet formation.

**Very poor dispersion--many lumps.

TABLE 11

EFFECT OF HEAT TREATMENT TIME AT 800°C ON FILTER STRENGTH*

Filter Sample	Min. at 800°C	Tensile Strength, lb/in.	
		Before 800°C	After 800°C
21-6A	5	0.12	0.11
21-6B	30	0.12	0.24
21-6C	60	0.12	0.21
21-6D	120	0.12	0.21

*Beating conditions: 1 min. at low speed.

(24 hrs at 425°C) were used. The fibers appeared well dispersed and of reasonable length, but did not increase sheet strength, as shown in Table 12.

B. EFFECT OF ALKALINE TREATMENT AND HEAT TREATMENT ON FILTER STRENGTH

To prevent silica fibers from sticking together in aqueous dispersions, it has been found necessary to use low pH (2.0 to 4.5). This effect is not well understood, but it suggested the possibility that high pH might somehow strengthen the filter. We therefore prepared several handsheets, as before, except that fibers were beaten at low speed from 1 to 3 minutes, and damp-dry filters were treated with various alkaline solutions and washed.*

Results, as shown in Table 13, indicate that treatment with 0.05 to 0.1N sodium hydroxide, followed by rinsing with distilled water and heating 30 minutes at 800°C produced the best filter. Strength (0.8 lb/in.), flexibility, and filtration performance (99% DOP efficiency at 320 cm/min) are all satisfactory.

Because all raw materials can be high purity, this treatment is considered highly promising and a major breakthrough in the program. The cost of this filter is expected to be satisfactory. As indicated in Section II.B, the cost of Johns-Manville 99.2% Microfibers is about $25/lb. Fiber cost would therefore be about $0.50/ft^2. Total filter cost should be about $1.00/ft^2.

The Table 13 data also indicate that potassium hydroxide, ammonium hydroxide, and tetramethylammonium hydroxide increase filter strength significantly, but not as much as sodium hydroxide. Sodium chloride does not increase strength at all. The lithium hydroxide results appear anomalous. Sodium carbonate should be further evaluated at lower concentrations.

To determine the effect of higher temperatures on filter strength, the 0.05N NaOH and rinse treatment was repeated except that three temperatures were used. Results, as shown in Table 14, indicate that 30 minutes at 800°C is better than 30 minutes at 870°C or 930°C. However, the

*Alkaline treatment and washing procedure is as follows:

After sheet formation by gravity draining, suck dry for 5 seconds and shut off vacuum pump. Pour 100 cc of alkaline solution carefully around the edge of the sheet to wet the entire sheet. Suck dry and shut off pump. Add another 100 cc and suck dry. Repeat this with 200 cc more (400 cc total). After sucking dry, wash the sheet with 1000 cc of distilled H_2O. The first 300 cc are added without suction (poured around the sheet so as not to upset the fiber mass). Suck dry, leave suction on, and pour the rest of the distilled H_2O directly on the sheet (in approximately 10-15 sec). Shut off pump; lift off sheet; place on blotters and dry 15 minutes on drum drier at 116°C. Heat treat at 800°C for 30 minutes.

TABLE 12

EFFECT OF LONG FIBERS (1/4" ASTROQUARTZ)
ON FILTER STRENGTH*

Sample No.	Astroquartz	Tensile Strength lbs/in.
12-A	None (control)	0.12
12-E	Silane coated**	0.11
12-F	Heat cleaned***	0.13

*Filter contained 40% Code 106 and 30% Code 108 Microfibers and 30% Astroquartz. Code 106 and Code 108 fibers were beaten 1 minute at low speed.

**Silane-coated Astroquartz (2 g) was agitated in a 1-qt Waring blender for 5 minutes at lower than normal speed with 400 g water at pH 2-2.5 (with HCl) and 0.01 g Daxad 11 (Dewey and Almy Chemical Co. surfactant). The dispersion was diluted to 3 ℓ, 10 cc of 0.5% aq. solution of Percol 156 (Allied Colloids, Inc., anionic polyacrylamide dispersing agent) added, and bucketed 25 times.

***Heat cleaned 1/4-in. Astroquartz (0.6 g) was agitated in 1-qt Waring blender 4 min. at low speed with 400 g water at pH 2-2.5 (with HCl) and 0.01 g Daxad 27.

TABLE 13

EFFECT OF ALKALINE TREATMENT* AND HEAT TREATMENT ON FILTER STRENGTH

Filter No.	Beating Time at Low Speed	Alkaline Solution	Rinse Water	Before 800°C Heat Treatment		After 30 min @ 800°C	
				Tensile Strength lb/in.	Comments	Tensile Strength lb/in.	Comments
18A-1	1	None	None	0.12	Control	0.33	Flexible
18B-1	1	1 N NaOH	None	0.40	Brittle, cannot be folded	0.40	Brittle, cracks when folded
18C-1	1	1 N NaOH	Dist H_2O	0.35	Flexible	0.75	Stiff, can be folded
18D-1	1	25% Na_2CO_3	None	0.55	Fairly flexible	--	Brittle, cracks when folded
18E-1	1	25% Na_2CO_3	Dist H_2O	0.20	Fairly flexible	0.50	Brittle, cracks when folded
21-2	1	1 N NaOH	pH 4 H_2O	0.23	Flexible	0.49	Flexible
21-3	1	1 N NH_4OH	Dist H_2O	0.28	Flexible	0.39	Flexible
21-4	1	0.1 N NaOH	None	0.35	Slightly flexible	0.72	Brittle
21-5**	1	0.1 N NaOH	Dist H_2O	0.33	Very flexible	0.82	Flexible
23A	3	0.05 N NaOH	Dist H_2O	0.34	Very flexible	0.83	Flexible
23B	3	0.01 N NaOH	Dist H_2O	0.20	Very flexible	0.48	Flexible
26-2	3	0.05 N KOH	Dist H_2O	0.26	Very flexible	0.51	Flexible
26-3	3	0.05 N $(CH_3)_4NOH$	Dist H_2O	0.20	Very flexible	0.52	Flexible
26-4	3	0.05N LiOH	Dist H_2O	0.31	Very flexible	0.08	Flexible
26-5	3	0.05N NaCL	Dist H_2O	0.34	Very flexible	0.33	Flexible

*Alkaline solution volume was 400 ml. Rinse water volume was about 1000 ml.

**Four additional Type 21-5 filters averaged 0.74 lb/in. tensile strength and were quite flexible. DOP efficiencies at 320 cm/min of 99.0 to 99.4% at 22 to 26 mm H_2O ΔP, respectively, were obtained.

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TABLE 14

EFFECT OF HEATING TEMPERATURES
ON FILTER STRENGTH
(30 min heating)

Filter No.	Temperature °C	Tensile Strength lbs/in.	Remarks
23-A	800	0.83	Flexible, foldable
25-4A	870	0.50	Brittle, non-foldable
25-4B	930	0.68	Brittle, non-foldable

effect of time on strength should also be determined.

The Gelman Instrument Co. provided us with two samples of quartz fiber filter paper made by W. & R. Balston, Ltd. We do not know whether these sheets were made by hand or machine, but understand that an inorganic binder has been added to one sample to improve strength. We treated the dry filters with 0.05N NaOH and rinsed and heated them, as described above, to determine the effect on strength. Results are shown in Table 15. All samples were flexible and foldable.

The low tensile strength of the untreated Gelman Type E Quartz suggests that the filter may have been damaged in the mail. Gelman's tensile data was much higher (about 0.26 vs. 0.08 lb/in.).

The Gelman Type F Quartz sample was difficult to wet during the alkaline treatment (suggesting a hydrophobic organic binder) but was strengthened substantially by heating (from 0.62 to 0.90 lbs/in.).

We also treated dry Pallflex "Tissuquartz" with 0.05N NaOH and rinsed and heated it, as described in Section B, to determine the effect on strength and filtration performance. Results are shown in Table 16. All samples were flexible and foldable. These results show that "Tissuquartz" can be substantially strengthened by this treatment (from 0.28 to 0.79 lbs/in.) without degrading filter performance.

To gain some insight into scale-up problems, we treated some Pallflex material in the same manner on a larger handsheet mold (12" x 12"). This mold is not equipped with a vacuum pump (as is the 6" mold) and drains by gravity only. It was difficult to apply the sodium hydroxide and wash water without damaging the sheet. The sheet had improved strength but much lower filtration performance, as shown in Table 17. The results confirm that the wet sheet was damaged by the application of the alkaline and wash liquids and suggest that the liquids be applied carefully with the sheet under suction.

C. ALKALINE TREATMENT AND HEAT TREATMENT WITHOUT WATER WASH

We investigated the effect of omitting the water wash by treating "Tissuquartz" filters with 0.001N and 0.002N sodium hydroxide. We applied 50 ml to a dry 6" diameter filter, pumped it damp-dry, and repeated the step. Each unwashed filter was then dried 15 minutes on a drum drier at 116°C and heated at 800°C. Results, as shown in Table 18, indicate that 0.002N NaOH and 30 minutes at 800°C produced high-strength filters (1.14 to 1.30 lbs/in.). All filters could be folded without cracking, indicating that higher NaOH concentrations without washing should be evaluated.

To confirm these results as well as to investigate the effect of slurry dilution and draining velocity, we prepared several slurry batches and formed sheets in the 6" diameter mold, as shown in Table 19.

TABLE 15

EFFECT OF ALKALINE TREATMENT AND HEAT TREATMENT ON GELMAN QUARTZ FIBER FILTERS

Filter No.	Type	Treatment	Wt 6 in. Diam Disc gms	Tensile Strength lbs/in.
24-I-EA	Gelman Type E Quartz	None	1.80	0.08
24-I-EB	Gelman Type E Quartz	0.05 N NaOH	--	0.09
24-I-EC	Gelman Type E Quartz	0.05" NaOH + 30 min at 800°C	--	0.42
24-I-FA	Gelman Type F Quartz	None	1.65	0.62
24-I-FB	Gelman Type F Quartz	0.05 N NaOH	--	0.80
24-I-FC	Gelman Type F Quartz	0.05 N NaOH + 30 min at 800°C	--	0.90

TABLE 16

EFFECT OF ALKALINE TREATMENT AND HEAT TREATMENT ON PALLFLEX "TISSUQUARTZ"

Filter	Tensile Strength lbs/in.	Filtration Properties @ 320 cm/min	
		% DOP Efficiency	Flow Resistance mm H_2O
As received	0.28	99.99	36
After treatment and washing	0.41	--	--
After 30 minutes at 800°C	0.79	99.99	38.5

TABLE 17

EFFECT OF NaOH APPLICATION
TO PALLFLEX "TISSUQUARTZ" ON 12" x 12" MOLD

Filter	Tensile Strength lbs/in.	Filtration Properties at 320 cm/min	
		% DOP Efficiency	Flow Resistance mm H_2O
As received	0.28	99.99	36
After treatment and washing	0.41	--	--
After 30 minutes at 800°C	0.62	97.8	21

TABLE 18

EFFECT OF OMITTING WATER WASH ON TENSILE STRENGTH OF PALLFLEX "TISSUQUARTZ"

NaOH Concentration	Time at 800°C (mins.)	Tensile Strength (lbs/in.)
0.001 N	15	0.60 to 0.80
"	30	0.90 to 0.91
"	60	0.83 to 0.92
0.002 N	15	0.72 to 0.77
"	30	1.14 to 1.30
"	60	0.75 to 0.89

TABLE 19

EFFECT OF SHEET FORMATION CONDITIONS ON TENSILE STRENGTH

Sample	Sheet Formation Conditions	Tensile Strength (lbs/in.)
1.	Gravity drain 2 l of slurry	0.12 to 0.14
2.	Mix 2 l of slurry with 2 l of pH2 tap water in mold and gravity drain	0.18 to 0.18
3.	Pump out 2 l of slurry	0.43 to 0.46
4.	Mix 2 l of slurry with 2 l of pH2 tap water in mold and pump out slurry	0.20 to 0.23*

*Sheet was nonuniform due to mold swirl.

In each case, we beat 1 g of 99.2% Code 106 Johns Manville old process "Microquartz" for 1 minute in 2 ℓ of tap water adjusted to pH 2 with HCl. Beating was done at low speed in a 6 ℓ stainless steel Waring blender. We then added 1 g of 99.5% Code 108 new process "Microquartz" and beat another minute. After formation, sheets were treated with 0.002N NaOH without washing as above, dried on a drum drier for 15 minutes at 116°C, and heated 30 minutes at 800°C. The results are shown in Table 19. All sheets could be folded without cracking. The results suggest that pumped drainage of dilute fiber slurries increases strength, and that more concentrated NaOH should be evaluated.

To evaluate the effect of NaOH concentration, additional slurries were prepared as above and drained by pumping. While still under suction, the sheets were treated with 500 ml of NaOH, pumped to damp dryness, and treated again. Unwashed samples were then dried on a drum drier for 15 minutes at 116°C and heated 30 minutes at 800°C. Results, as listed in Table 20, indicate that NaOH concentration should not exceed 0.005N without washing.

D. EFFECT OF FIBER SIZE AND PURITY ON TENSILE STRENGTH

Previous handsheet samples with adequate strength have all been made with 99.2% Johns-Manville old process Code 106 and Code 108 "Microquartz." To investigate the effect of fiber size and purity on tensile strength, we prepared the fiber slurries shown in Table 21. In each case, we beat 2 g of fibers for 2 minutes in 2 ℓ of tap water adjusted to pH 2 with HCl. Beating was done at low speed in a 6 ℓ stainless steel Waring blender. Sheets were drained by pumping and treated with 500 ml of 0.002N NaOH with suction as above (twice). Unwashed sheets were dried on a drum drier for 15 minutes at 116°C and heated 30 minutes at 800°C.

Table 21 indicates that 99.5% Code 108 new process "Microquartz" produces weaker sheets than 99.2% Code 108 old process "Microquartz." This may result from excessive leaching and weakening of the 99.5% fibers. However, these fibers should be further evaluated. Table 21 also indicates that the larger Code 112 fibers reduce sheet strength.

The high strength of some of these sheets is attributed to the beating and forming conditions. The stronger sheets in Table 21 cracked very slightly when folded, indicating that NaOH concentrations should not exceed 0.002N when sheets are prepared in this way.

E. SPRAY APPLICATION OF SODIUM HYDROXIDE AND SODIUM CARBONATE TO PALLFLEX "TISSUQUARTZ"

In work thus far, NaOH has been applied to filter handsheets by pouring from a beaker. To evaluate spray application of alkali, we placed Pallflex "Tissuquartz" on a dry 6-inch diameter handsheet mold under suction and sprayed it with 0.002 N NaOH in tap water for various times. We used an H. D. Hudson* 2-gallon "Clipper" 6215 galvanized sprayer with

*403 E. Illinois Street, Chicago, Illinois 60611

TABLE 20

EFFECT OF NaOH CONCENTRATION AND APPLICATION TO SHEET UNDER SUCTION

NaOH Concentration	Tensile Strength (lbs/in.)	Remarks
0.002 N	0.38 to 0.49	Foldable without cracking*
0.005 N	1.50 to 1.73	Brittle
0.01 N	0.69 to 1.41	Very brittle

*DOP penetration 0.67 to 0.95% at 850 cm/min.
Flow resistance 72 to 76 mm H_2O at 850 cm/min.

TABLE 21

EFFECT OF FIBER SIZE AND PURITY ON TENSILE STRENGTH

Sample	Composition				Tensile Strength (lbs/in.)
	99.2% Code 106 (0.5 to 0.7 μm dia)	99.2% Code 108 (0.7 to 1.6 μm dia)	99.5% Code 108 (0.7 to 1.6 μm dia)	98.5% Code 112 (2.6 to 3.8 μm dia)	
1.	1 g		1 g		0.65 to 0.97
2.	1 g	1 g			1.12 to 1.18
3.	1 g			1 g	0.33 to 0.52
4.	2 g				0.84 to 1.16

Cone Nozzle Cap, No. 113-476. Spray rate was about 400 ml/min. Filters were kept under suction until damp dry and were not washed. They were then dried on a drum drier for 15 min at 116°C and heated 30 min at 800°C. Results are shown in Table 22.

These results suggest that 15 seconds of spraying is adequate, that even shorter spray times may be satisfactory, and that each sheet was uniformly covered with NaOH.

The different strengths of duplicate sheets may be due to different degrees of damp dryness of the sheets, caused by poor design of our handsheet mold screen. The screen is supported by a rather thick perforated plate which retains liquid by capillary action. When the suction is shut off after damp-drying the handsheet, the sheet is rewetted by the residual water in the support plate. The amount of rewetting appears to be somewhat variable. We therefore are making a new handsheet mold to eliminate this problem.

We also sprayed dry "Tissuquartz" filters for 30 sec with sodium carbonate solutions of varying concentration. Results, as listed in Table 23, suggest that 0.002N to 0.005N Na_2CO_3 is preferable to 0.01 to 0.02N.

F. TENSILE STRENGTH VS. FIBER SIZE AND PROCESS CONDITIONS WITH SPRAYED NaOH

Previous results suggested that beating of fibers for 2 minutes followed by pumped drainage of dilute slurries and the NaOH and heat treatment would produce filters of high tensile strength (1 lb/in.). We therefore prepared several handsheets to determine the optimum beating time and slurry concentration.

In each case, we beat 1 g of 99.2% old process Code 106 fiber and 1 g of 99.2% old process Code 108 fiber in 2 ℓ of tap water adjusted to pH 2 with HCl. Beating was done at low speed in a 6 ℓ stainless steel Waring blender. Slurries were used either as is or diluted with 2 ℓ of tap water adjusted to pH 2 with HCl. Sheets were formed by pumped drainage, sucked to damp dryness, sprayed for 30 sec with 0.002N NaOH, and sucked again to damp dryness. Unwashed sheets were then dried on a drum drier for 15 min at 116°C and heated 30 min at 800°C. Results are listed in Table 24.

These results indicate an adverse effect on dioctyl phthalate (DOP) smoke penetration of beating for 5 minutes and dilution of slurry (samples 3 and 9), and an adverse effect on flexibility of beating for 3 minutes (sample 7). They suggest that the beating time and slurry volume under these conditions should be 1 to 2 minutes and 2 liters, respectively, and that 0.001N NaOH may improve flexibility.

Similar experiments were also performed with all Code 106 fiber, except that two slurries were gravity drained rather than pumped out of the handsheet mold. Results are listed in Table 25.

TABLE 22

EFFECT OF 0.002N NaOH SPRAY TIME ON TENSILE STRENGTH OF PALLFLEX "TISSUQUARTZ"

Sample	Spray Time (sec)	Tensile Strength (lbs/in.)	Cracks When Folded?
1	15	0.74 to 0.76	No
2	15	1.17 to 1.27	No
3	15	1.34 to 1.37	Yes
4	30	0.99 to 1.01	No
5	30	0.90 to 0.93	No
6	45	1.02 to 1.12	No
7	45	1.05 to 1.09	No
8	60	0.71 to 0.72	No
9	60	1.32 to 1.38	Slightly

TABLE 23

EFFECT OF Na_2CO_3 CONCENTRATION
ON TENSILE STRENGTH OF PALLFLEX "TISSUQUARTZ"

Sample	Na_2CO_3 Concentration	Tensile Strength (lbs/in.)	Cracks When Folded?
1	0.002N	0.96 to 1.01	No
2	0.002N	0.93 to 1.02	No
3	0.005N	0.82 to 1.00	No
4	0.005N	0.97 to 1.00	No
5	0.01N	0.85 to 0.97	No
6	0.01N	0.80 to 0.87	Slightly
7	0.02N	0.87 to 0.92	Slightly
8	0.02N	1.04 to 1.06	Slightly

TABLE 24

EFFECT OF BEATING TIME AND FIBER SLURRY CONCENTRATION ON TENSILE STRENGTH OF 50% CODE 106/50% CODE 108 "MICROQUARTZ" HANDSHEETS

Sample	Beating Time (min)	Slurry Volume (liters)	DOP Penetration at 320 cm/min (%)	Flow Resistance at 320 cm/min (mm H_2O)	Tensile Strength (lbs/in.)	Cracks When Folded?
1	2	2	0.34	29	1.27 to 1.27	Slightly
3	2	4	0.67	26	1.66 to 1.72	Yes
5	1	2	0.27	28.8	1.17 to 1.36	Slightly
7	3	2	0.26	30	1.72 to 1.90	Yes
9	5	2	1.15	25.5	1.62 to 1.76	Yes

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TABLE 25

EFFECT OF BEATING TIME AND SHEET FORMATION CONDITIONS
ON TENSILE STRENGTH OF 100% CODE 106 "MICROQUARTZ" HANDSHEETS

Sample	Beating Time (min)	Sheet Formation Conditions	Tensile Strength (lbs/in.)	Cracks when Folded?	Remarks
2	2	Pump out 2 l of slurry	0.70 to 0.94	Yes	Sheet stuck to screen.
4	2	Pump out 4 l of slurry	--	--	Sheet had pinholes and stuck tightly to screen.
6	1	Pump out 2 l of slurry	0.63 to 0.83	Yes	Sheet stuck to screen.
8	3	Gravity drain 2 l of slurry	--	--	Sheet stuck tightly to screen.
10	1	Gravity drain 2 l of slurry	0.72 to 1.07	Yes	Sheet stuck to screen.

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These results also suggest that beating time should be 1 to 2 minutes, that the slurry should not be diluted to 4 liters, and that 0.001N NaOH may improve flexibility. The pinhole formation in the diluted slurry sample (#4) may explain the increased DOP penetration of the diluted slurry sample (#3) in Table 24.

A comparison of Tables 24 and 25 suggest that Code 106 fibers be blended with at least 10% Code 108 fibers to prevent sticking to the handsheet mold screen, and increase strength and flexibility.

G. SPRAY APPLICATION OF 70% CODE 106/30% CODE 108 "MICROQUARTZ" FILTERS WITH NaOH AND Na_2CO_3

To confirm the above indications, we prepared several handsheets as above except that:

1. Fibers were 70% of 99.2% old process Code 106 "Microquartz" and 30% of 99.2% old process Code 108 "Microquartz."

2. Beating time was 2 minutes.

3. Slurries were used as is (2 liters volume).

4. Heating time was varied from 30 to 120 minutes. Results are listed in Table 26.

These results suggest that 0.001N NaOH is too dilute. None of the sheets cracked when folded, but tensile strength was low. The results confirmed previous indications that pumped drainage gives stronger filters than gravity drainage, and suggest that heating for 60 minutes at this low concentration maximizes strength.

We also prepared several handsheets as above except that 0.002N and 0.005N Na_2CO_3 was used instead of NaOH. Results, as given in Table 27, agree with the indications in Table 26 that heating for 60 minutes at this low concentration maximizes strength.

The gravity-drained 0.002N Na_2CO_3 sprayed filters in Table 27 (samples 5 through 8) appear somewhat anomalous in that they are generally stronger than the pump-drained filters (samples 1 through 4) and one sample (#8) is much stronger and more brittle than would be expected. These results may be due to the handsheet screen problem discussed in Section IV.E.

In general, these results indicate that handsheet filters with satisfactory efficiency, temperature resistance, cost, strength, and flexibility can be made by dilute aqueous alkali spraying and heating. The effect of the alkaline treatment on purity, moisture sensitivity, pH, etc. should now be evaluated. The scale-up of the strengthening treatment to machine production of filter media should also be evaluated.

TABLE 26

TENSILE STRENGTH OF 70% CODE 106 AND 30% CODE 108
"MICROQUARTZ" HANDSHEETS SPRAYED WITH 0.001N NaOH

Sample	Type of Handsheet Mold Drainage	Time at 800°C (min)	Tensile Strength (lbs/in.)	Cracks When Folded?
1	Pumped	30	0.26 to 0.32	No
2	Pumped	60	0.51 to 0.57	No
3	Pumped	90	0.32 to 0.35	No
4	Pumped	120	0.36 to 0.38	No
5	Gravity	30	0.22 to 0.23	No
6	Gravity	60	0.42 to 0.46	No
7	Gravity	90	0.23 to 0.27	No
8	Gravity	120	0.43 to 0.44	No

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TABLE 27

TENSILE STRENGTH OF 70% CODE 106/30% CODE 108
"MICROQUARTZ" HANDSHEETS SPRAYED WITH Na_2CO_3

Sample	Na_2CO_3 Concentration	Type of Handsheet Mold Drainage	Time at 800°C (min)	Tensile Strength (lbs/in.)	Cracks When Folded?
1	0.002N	Pumped	30	0.34 to 0.38	No
2	0.002N	Pumped	60	0.47 to 0.53	No
3	0.002N	Pumped	90	0.48 to 0.56	No
4	0.002N	Pumped	120	0.56 to 0.56	No
5	0.002N	Gravity	30	0.58 to 0.62	No
6	0.002N	Gravity	60	0.57 to 0.61	No
7	0.002N	Gravity	90	0.33 to 0.35	No
8	0.002N	Gravity	120	1.13 to 1.17	Yes
9	0.005N	Pumped	30	1.34 to 1.44	Slightly
10	0.005N	Pumped	60	1.21 to 1.24	Slightly
11	0.005N	Gravity	30	1.10 to 1.15	Slightly
12	0.005N	Gravity	60	0.66 to 0.77	No

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V. HANDSHEET FILTER CHARACTERISTICS

A. PRODUCTION OF HIGH PURITY HANDSHEETS

One hundred high-purity 6-inch diameter handsheets were prepared with 99.2% old process "Microquartz" fibers for filter characterization purposes, as follows:

No. of Sheets	% Code 106 "Microquartz"	% Code 108 "Microquartz"
40	90	10
20	70	30
40	50	50

Special care was taken to achieve high purity, including the fabrication and use of an improved 6-1/2-inch diameter handsheet mold. This mold was all stainless steel except for a very small carbon steel area which was painted with zinc chromate primer and coated with Pliobond rubber adhesive. In each case, 2 grams of fiber and 2 ℓ of distilled water, adjusted to pH 2 with Fisher reagent grade HCl, were beaten for 2 minutes at low speed in a 6 ℓ stainless steel Waring blender. The slurry was then poured into the stainless steel handsheet mold and drained by pumping.

The formation screen was a Millipore Corporation 293 mm diameter stainless steel support screen with 0.005" diameter holes spaced 0.005" apart. To minimize filter contamination, the mold was not filled with water to screen level before the slurry was added, as had been done previously.

Half of the above filters were sprayed under suction with 0.005 N NaOH and half with 0.005 N Na_2CO_3 (both Fisher Certified reagent solutions) in distilled water. The filters were sprayed for 15 seconds at a flow rate of 250 ml/min with a Millipore stainless steel fan spray gun at a pressure of 15 psi. The solutions were pressurized in a Sears Roebuck stainless steel garden sprayer in which the brass air pump was replaced with a connection to compressed nitrogen. The formation screen was blown free of loose fibers with compressed nitrogen before each sheet was made. The filters were damp-dried without rinsing, placed between blotters lined with Whatman No. 1 analytical filter paper and dried on a drum drier at 115°C for 15 minutes.

The filters were then placed in stacks of 10 on a thin sheet of Hastelloy and heated 1 hour at 800°C. The top and bottom filters were considered contaminated from the Hastelloy and possible dusting from the furnace lining, and were used for DOP and tensile tests. The remaining filters were trimmed to 5-3/4" diameter, placed between Whatman No. 1 filter papers, and sealed in polyethylene envelopes. These filters averaged

about 1.38 g, which is 7.6 g/ft^2 or 4.2 g/80 in.^{2}. Fiber loss in processing was about 20%.

B. FILTRATION EFFICIENCY, FLOW RESISTANCE, AND TENSILE STRENGTH

DOP and tensile test data were obtained on samples of the 100 handsheets. Results were highly variable, as shown in Table 28. The variability is attributed to use of the new stainless steel handsheet mold and alkali spraying system without thoroughly evaluating their performance. Although less variability would have been preferred, the filters are satisfactory for the measurements for which they are intended (chemical analysis, pH measurements, effects of humidity on filter weight, and filter efficiency tests).

Despite the variability, the data in Table 28 indicate that filter efficiency can be increased by using less Code 108 "Microquartz" and more Code 106 "Microquartz." Table 28 also suggests that the higher efficiency filters are slightly stronger than those of lower efficiency and that the sodium carbonate-treated filters are slightly stronger than those treated with sodium hydroxide.

The effect of filtration velocity on DOP penetration and flow resistance of 10% Code 108 and 30% Code 108 "Microquartz" filters is shown in Figures 1 and 2, respectively. Note that penetration reaches a maximum at about 40 cm/sec and that flow resistance increases linearly with velocity.

We generated 4.5 μm and 12 μm monodisperse sodium chloride particles with the Berglund-Liu aerosol generator and attempted to perform filter penetration tests with them by light scattering. However, the particle concentrations were too low to be measured by this technique.

Efficiency of collection of solid, high-density particles has not been measured, but we expect the 99.9% DOP efficiency filters to be at least 99% efficient on such particles.

C. EFFECT OF HUMIDITY ON FIBER FILTER WEIGHT

When used for gravimetric analysis, sampling filters should not change weight at different humidities. To evaluate this effect, we conditioned and weighed both the NaOH and Na_2CO_3-treated 10% Code 108 filters in Table 28 and Gelman A glass fiber filters after conditioning overnight, using the following sequence:

1. At 105°C
2. Over saturated calcium chloride (32% RH)
3. Over Drierite
4. Over saturated calcium chloride (32% RH)

TABLE 28

PHYSICAL PROPERTIES OF HIGH-PURITY HANDSHEET SAMPLES PREPARED ON STAINLESS STEEL EQUIPMENT

% Code 108 "Microquartz"	No. of Samples	0.005N Alkali	DOP Penetration at 320 cm/min. (%)	Flow Resistance at 320 cm/min. (mm H_2O)	Tensile Strength lbs/in.
10	4	NaOH	0.01 to 0.04	37 to 43	0.18 to 0.63
10	3	Na_2CO_3	0.005 to 0.04	29 to 48	0.35 to 0.88
30	2	NaOH	0.20 to 0.34	25 to 28	0.17 to 0.42
30	2	Na_2CO_3	0.04 to 0.43	24.5 to 34.5	0.26 to 0.67
50	4	NaOH	0.26 to 1.10	19.5 to 24	0.14 to 0.37
50	4	Na_2CO_3	0.15 to 0.73	22 to 27	0.13 to 0.49

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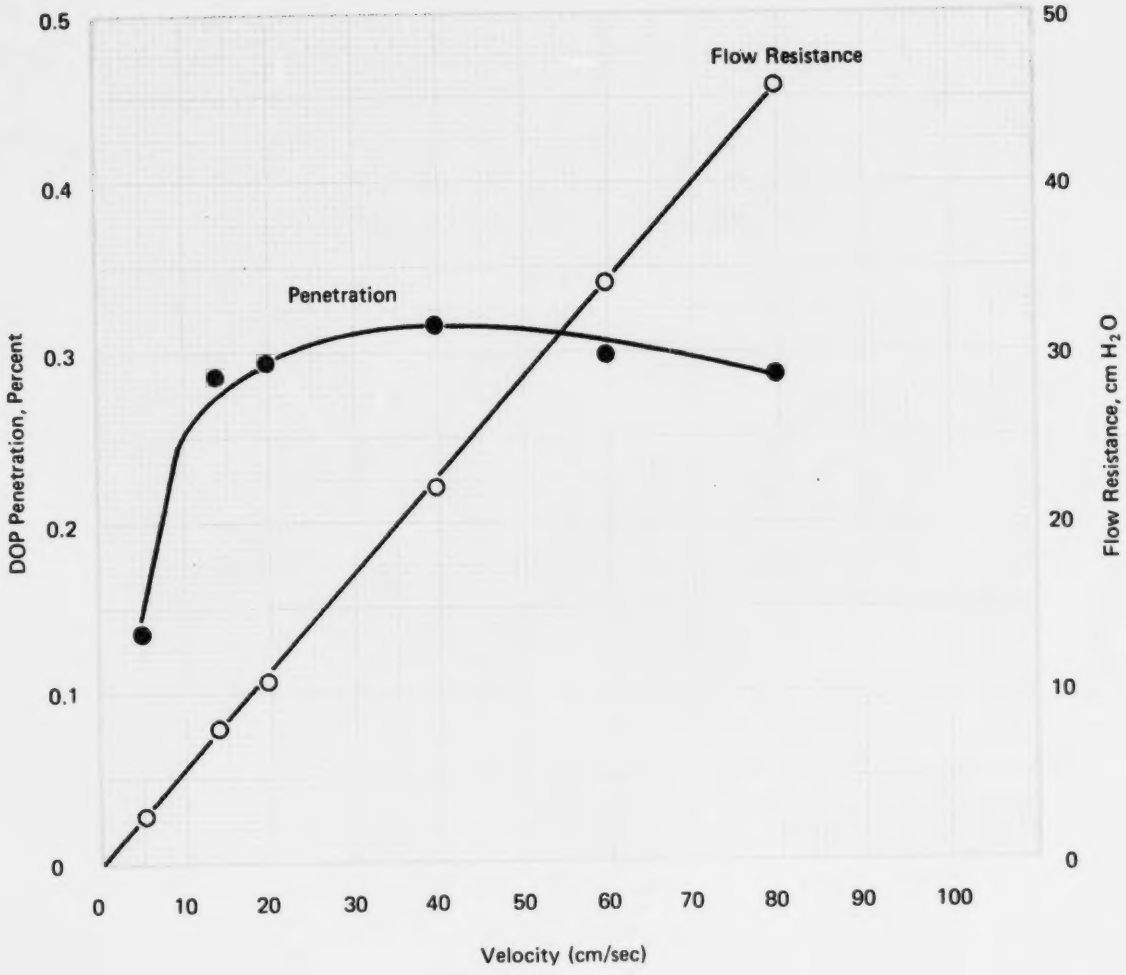

FIGURE 1 DOP PENETRATION AND FLOW RESISTANCE VERSUS VELOCITY OF 30% CODE 108/70% CODE 106 "MICROQUARTZ" FILTER STRENGTHENED WITH 0.005N NaOH

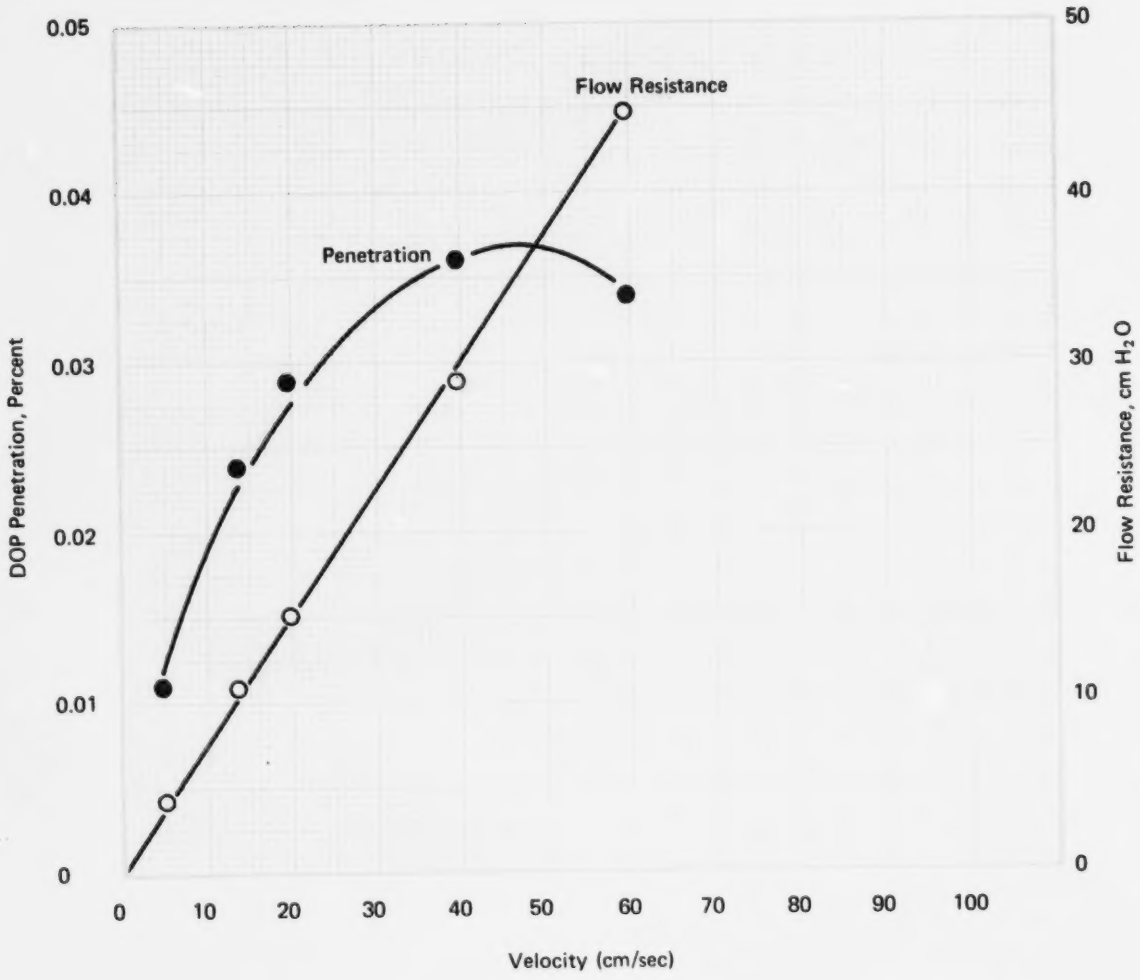

FIGURE 2 DOP PENETRATION AND FLOW RESISTANCE VERSUS VELOCITY OF 10% CODE 108/90% CODE 106 "MICROQUARTZ" FILTER STRENGTHENED WITH 0.005 N NaOH

Tests were performed in duplicate, and average weight changes relative to oven-dry weights are shown in Figure 3. None of the filters was significantly water-sensitive; all weight changes were less than 0.2%.

In a separate but similar experiment, we compared the effects of humidity on 2 glass fiber filters and 2 Pallflex "Tissuquartz" filters. One "Tissuquartz" filter had been strengthened by the sodium hydroxide and heat treatment described in Section IV.C. Results, as shown in Figure 4, indicate that "Tissuquartz" is much more water-sensitive than the other filters (up to 7% weight change). The reduced sensitivity of the strengthened "Tissuquartz" (similar to glass fiber filters) is attributed to pore closing by heat treatment.

The experimental technique used in these experiments is not good enough to permit direct comparison of the previous humidity effects with these results (filters were exposed to room air during weighings). However, the relative performance in each set of experiments is believed to be significant. We conclude that either NaOH or Na_2CO_3-strengthened filters are highly insensitive to water vapor (similar to Gelman A glass filters).

D. FILTER pH

The alkalinity of sampling filters is believed to be important for gravimetric analysis due to possible filter reaction with acid gases. Therefore, although not a contract requirement, we measured the pH of several filter slurries. Slurries were prepared by stirring 2 g of filter in 100 ml of distilled water for 10 minutes. Slurry pH was measured when fresh and after standing 40 hours in watchglass-covered beakers. Results are given in Table 29.

Pallflex "Tissuquartz" has the lowest pH (7.2). Strengthened "Microquartz" filters are similar to glass (pH 8.3 to 10.1). These results suggest that the alkaline strengthening treatment may need modification to reduce filter pH where this is an important consideration.

E. SOLUBILITY IN ACID, ALKALINE, AND ORGANIC MEDIA

The "Microquartz" filters described in Section A above are highly insoluble in most acids and organic solvents, but are expected to be soluble in hydrofluoric acid, hot phosphoric acid, and aqueous alkali. They will be insoluble in organic solvents due to the use of all inorganic materials and a final 1 hour heat treatment at 800°C. Solubility in hot, dilute aqua regia is discussed in the following section.

F. EXTRACTABLE FILTER IMPURITIES

In Section III, we used emission spectrography to estimate the impurities in extracts of 98.5% and 99.2% "Microquartz" fibers and machine-made 98.5% "Microquartz" filters. These results indicated that the major

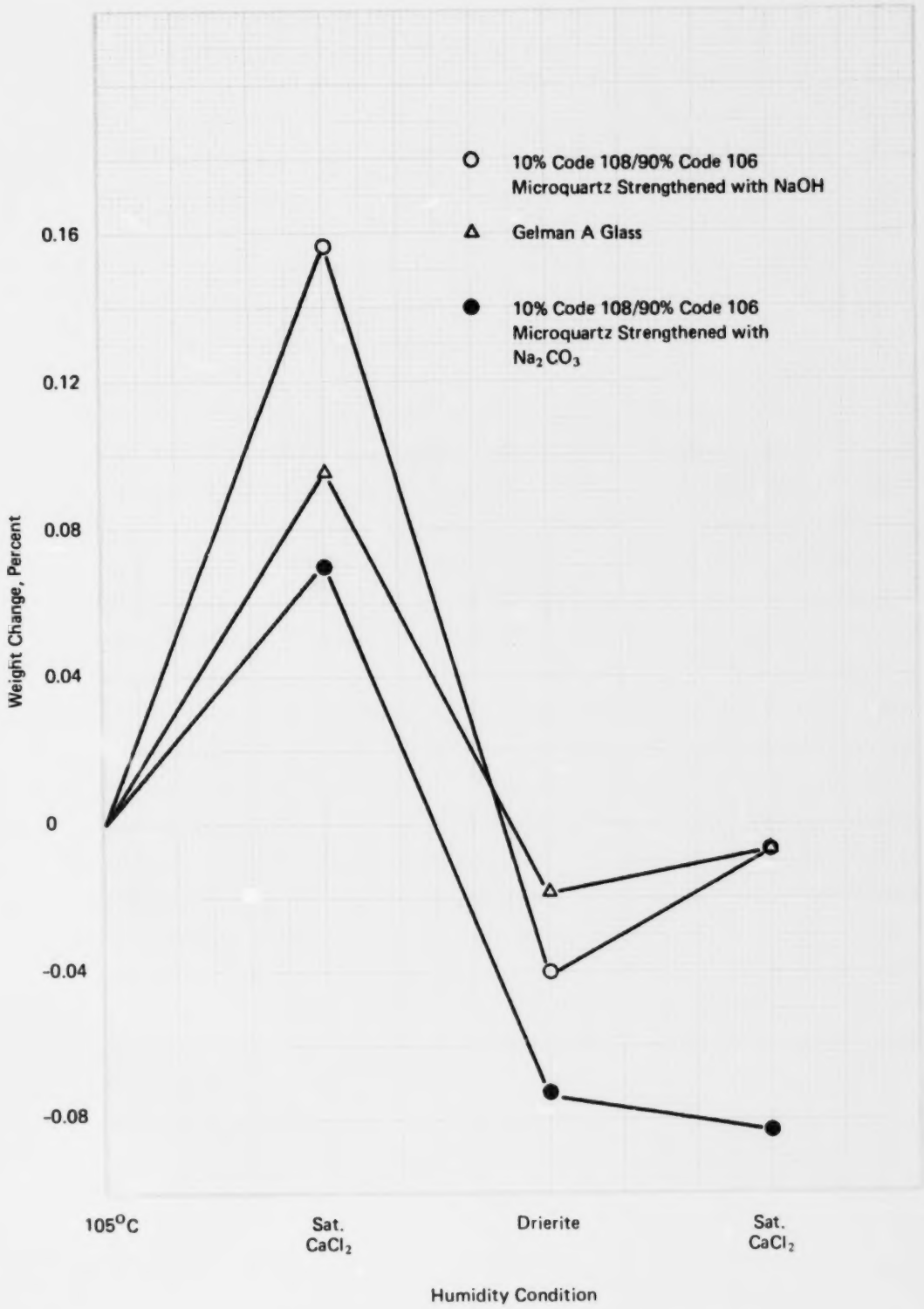

FIGURE 3 EFFECT OF HUMIDITY ON FILTER WEIGHT

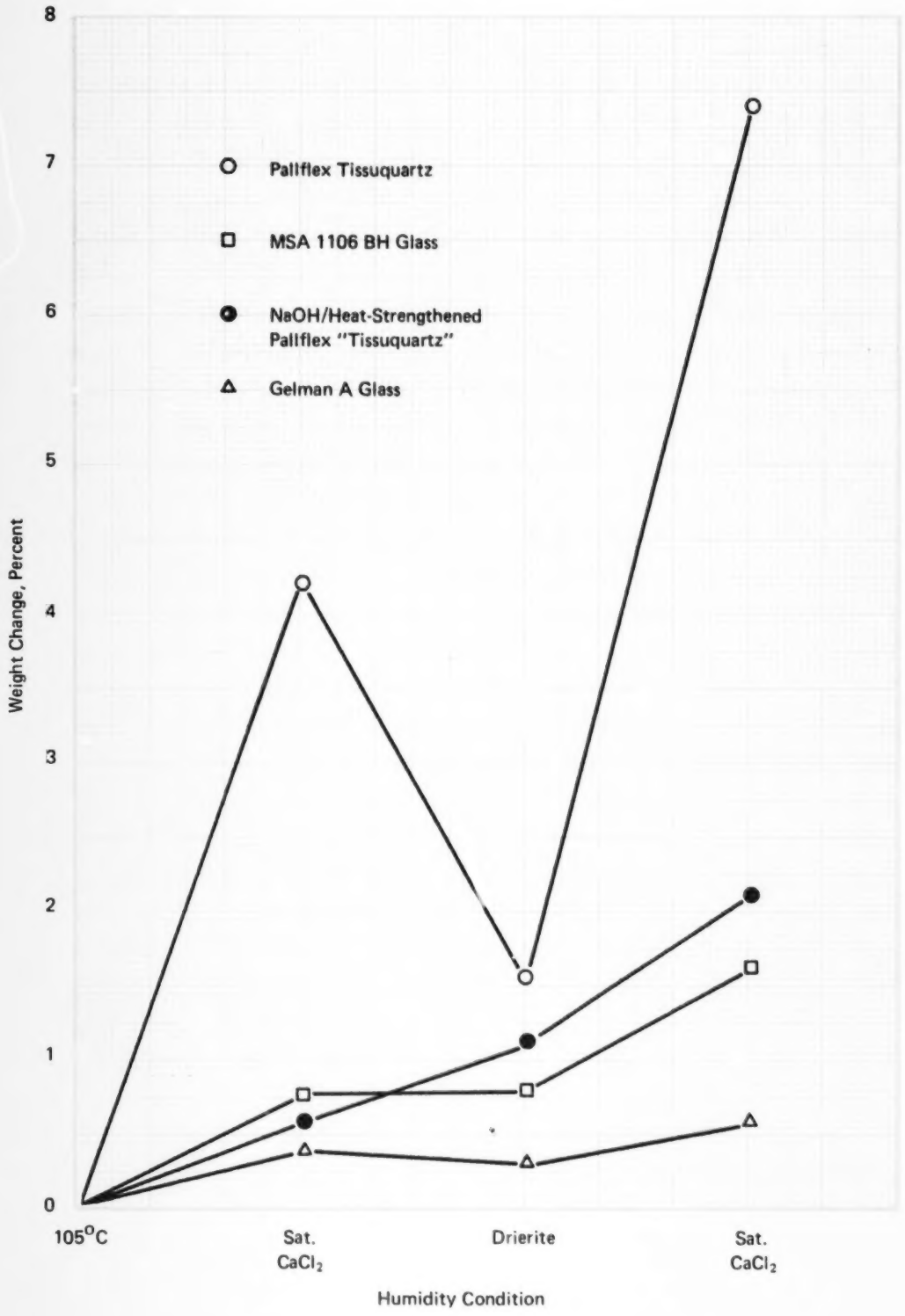

FIGURE 4 EFFECT OF HUMIDITY ON FILTER WEIGHT

TABLE 29

FILTER SLURRY pH

Filter	pH of Fresh Filter Slurry	pH of Filter Slurry after 40 hrs
Gelman A glass	8.3	7.0
MSA 1106 BH glass	10.0	8.7
Pallflex "Tissuquartz"	7.2	5.8
10% Code 108/90% Code 106 Filter treated with 0.05N NaOH and rinsed with distilled water*	9.3 to 9.8	7.3 to 7.4
10% Code 108/90% Code 106 Filter treated with 0.005N NaOH without rinse*	10.1	8.0
10% Code 108/90% Code 106 Filter treated with 0.005N Na_2CO_3 without rinse*	8.6 to 10.0	7.0 to 7.7

*"Microquartz"

extractable impurities of interest in 99.2% "Microquartz" filters will be iron and lead, and that aluminum, chromium, and titanium may also be above minimum detection levels. We therefore extracted and analyzed several of the 100 "Microquartz" 99.2% filters for these and several other metals by atomic absorption spectrometry.

A stack of eight trimmed handsheet filters (5.75" diameter) of the 10% Code 108 and 50% Code 108 "Microquartz" types listed in Table 28 was bisected to give duplicate samples weighing about 5.5 g each. These were extracted with a total of 240 ml of hot, dilute aqua regia (90 ml of 70% HNO_3, 20 ml of 37% HCl, 90 ml deionized water) for three hours. The extract was decanted off and the fibers washed with a few ml of 1:1 HCl. Extracts plus washings were evaporated nearly to dryness and then diluted to 100 ml with deionized water. Two reagent blanks were carried along with the samples. The results are presented in Table 30.

These results indicate that almost all purity requirements for optimum AAS and FES analysis have been achieved. Major extractable elements above the AAS and FES detection limits are lead, iron, aluminum, chromium, titanium, and zinc.

Aluminum, copper, iron, and lead levels are about the same in both the raw materials and the finished filters, but zinc levels are much higher in the finished filters. The zinc may have been extracted from the small Pliobond rubber-coated zinc chromate-painted area of the handsheet mold.

Table 30 also indicates that the extractable impurity levels are about the same in both NaOH and Na_2CO_3-strengthened filters and in both 10% Code 108 and 50% Code 108 "Microquartz" filters.

Because the "Microquartz" filters are expected to be used in applications where the purity of glass fiber filters is inadequate, it is of interest to compare the extractable filter impurities in these two materials. This is difficult to do because glass filters are made from different types of glass and filter purity varies widely from lot to lot. In general, however, emission spectrographic analyses of several MSA 1106BH and Gelman A glass fiber filters indicate that they have about ten times as much of at least one of the following extractable impurities as the "Microquartz" filters in Table 30:

Cr, Cu, Fe, Mn, Ni, Ti, Zn.

Lead levels in the several glass filters were about 50% higher than in the "Microquartz" filters.

G. SUITABILITY OF FILTERS FOR X-RAY FLUORESCENCE ANALYSIS AND NEUTRON ACTIVATION ANALYSIS OF COLLECTED PARTICULATES

Detailed analyses were not carried out using these techniques, but EPA was able to arrange for the following preliminary evaluations:

TABLE 30

MAJOR EXTRACTABLE IMPURITIES BY AAS IN 99.2% SILICA FIBERS AND FILTERS

Element	Minimum Detection Level (µg/g) AAS	FES	"Microquartz" Fiber Code 106	Code 108	10% Code 108/90% Code 106 "Microquartz" Filter NaOH Strengthened	Na_2CO_3 Strengthened	50% Code 108/50% Code 106 "Microquartz" Filter NaOH Strengthened	Na_2CO_3 Strengthened
Aluminum	1.2-12	0.2-2		22	26	26	13	25
				23	31	52	19	26
Chromium	0.2-2	0.2-2			3.0	6.3	2.7	4.3
					3.4	6.6	3.1	4.8
Copper	0.2-2	0.3-3	<0.8	<0.8	0.28	0.55	0.55	<0.08
					0.36	0.56	0.65	<0.08
Iron	0.2-2	1.6-1.6		8	9.8	11.1	6.7	0.5
				15	13.5	13.2	12.4	17.3
Lead	0.9-9	6.0-60	5.5	11	4.1	4.6	8.8	9.9
			<12	13	6.2	5.2	11.2	15.9
				18				
Magnesium	0.09-0.9	0.2-2			1.3	1.5	1.1	1.0
					1.4	2.2	1.2	1.3
Manganese	0.06-0.6	0.2-2			<0.4	<0.4	<0.4	<0.4
					<0.4	<0.4	<0.4	<0.4
Nickel	0.2-2	0.9-9			<2	<2	<2	<2
					<2	<2	<2	<2
Titanium	3.0-30	6.0-60			<20	<20	<20	<20
					<20	<20	<20	<20
Zinc	0.06-0.6	0.06-0.6	<0.25	<0.25	8.7	2.8	1.4	5.2
					10.1	3.7	1.8	7.0
Sodium	0.06-0.6	0.01-0.1			430	640	260	370
					550	650	280	390

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1. X-ray Fluorescence

Several of the 100 handsheets were analyzed by the sponsor on an energy dispersive X-ray fluorescence spectrometer made by the Lawrence Berkeley Laboratory, using evaporated foil standards. Samples were counted for 300 seconds, utilizing three secondary X-ray fluorescers. Results are listed in Table 31.

These results indicate that lead and iron are major impurities (1000 to 3000 ng/cm^2) and that these impurity levels increase with coarse fiber (Code 108) content. This finding is consistent with extractable fiber impurities (Table 5) and the fiber purification method (leaching), and suggests that the fine fiber content should be maximized. Impurities in the 100 to 1000 ng/cm^2 range are Ti, Ca, Sr, and Cr.

Identical samples were also analyzed in a preliminary way by Columbia Scientific Industries on their energy dispersive Model 110 Automated X-ray Fluorescence Analyzer. The general background for all filters was about 10% less than that from Whatman 41 paper, and the impurities were much lower than in glass fiber filters. Again, lead and iron were major impurities and these levels increased with coarse fiber content (up to 5000 ng/cm^2).

The EPA and Columbia results both indicate that the "Microquartz" filters are highly promising for X-ray fluorescence analysis and should be further evaluated. This evaluation should include the addition of standard particulate impurities at various depths in the filter.

2. Neutron Activation

Two of the 100 handsheets were analyzed by neutron activation by the sponsor. Samples were irradiated in the North Carolina State Pulstar Reactor at a flux of 10^{13} neutrons/cm^2/sec. One portion of each was analyzed directly by gamma ray spectrometry and the other was first subjected to radiochemical separation. Results are listed in Table 32.

These results are roughly consistent with the X-ray fluorescence analyses which indicate that Pb and Fe are major impurities. Pb is not detected by neutron activation, but Fe was detected at about 10X the concentration of any other impurity reported by this method.

The total Fe level (205-274 ppm) and the total Mn level (10-11 ppm) are at least 20X the extractable levels detected by AAS (Table 30). Total Cr and Zn levels measured by neutron activation are two or more times the extractable levels. These results confirm that dissolution of particulates from a filter for atomic absorption or flame emission analysis eases the requirements for filter purity.

We expected that sodium and aluminum might interfere with the neutron activation analyses but they were not mentioned in the EPA report. Extractable levels of these elements were measured by AAS to be about

TABLE 31

"MICROQUARTZ" FILTER IMPURITIES MEASURED BY X-RAY FLUORESCENCE (ng/cm^2)*

Element	10% Code 108/90% Code 106 "Microquartz" Filter Strengthened with Na_2Co_3	30% Code 108/70% Code 106 "Microquartz" Filter Strengthened with NaOH	50% Code 108/50% Code 106 "Microquartz" Filter Strengthened with Na_2CO_3
S	<300	<300	<200
Cl	<120	<150	<120
K	<100	<100	<100
Ca	170 ± 40	235 ± 60	150 ± 50
Ti	490	430	500
V	<30	<30	<30
Cr	70 ± 20	64 ± 30	150 ± 80
Mn	<30	<50	40 ± 30
Fe	1160	955	1360
Ni	36 ± 9	28 ± 8	39 ± 6
Zn	30 ± 15	25 ± 10	36 ± 10
Cu	25 ± 20	<30	<30
Br	<18	<20	<20
Pb	890	1930	3150
Se	<20	<20	<20
Ba	<250	<300	<250
As	<20	<20	<40
Rb	25 ± 15	<25	<25
Sr	180	160	162
Cd	<120	<150	<100

*Not corrected for attenuation. Errors ±15% in addition to those listed.

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TABLE 32

"MICROQUARTZ" FILTER IMPURITIES
MEASURED BY NEUTRON ACTIVATION ANALYSIS (ppm)

Element	10% Code 108/90% Code 106 "Microquartz" Filter Strengthened with NaOH	10% Code 108/90% Code 106 "Microquartz" Filter Strengthened with Na_2CO_3
Hg	0.02	--
As	0.05	1.0
Mn	9.7	11
Sb	15	11
Cr	16	26
Zn	19	10
Fe	205	274
Sc	0.10	0.10
Co	2.4	2.8

500 ppm Na and 25-50 ppm Al (Table 30).

The suitability of these filters for neutron activation analysis is uncertain and should be further evaluated, especially for purely instrumental analysis (no radiochemical separation). As recommended above for X-ray fluorescence analysis, additional evaluation should include the addition of standard particulate impurities. Various irradiation times and "cooling" times should also be included.

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APPENDIX

HIGH-EFFICIENCY GLASS FIBER FILTER PERFORMANCE CHARACTERISTICS

INTRODUCTION

High-efficiency glass fiber filters are commonly used to collect particulates from both ambient air and stack gases. The filter material was developed for the Atomic Energy Commission[1,2] for ventilation applications. It is a felt-like material composed of a combination of glass fibers in mixed sizes (about 0.5 to 3μm).

There are specifications for this filter for processing use (MIL-F-51079A) but not sampling applications. At the present time, two binderless filters (MSA 1106BH and Gelman Type A) have been found the most acceptable for atmospheric sampling and analysis[3,4] and are specified for use in the EPA particulate stack sampling train.

Because significant fractions of particulates collected in the EPA sampling train have been found in the impingers downstream of the filter, the efficiency of the filter has been questioned as follows:

- Is the filter efficiency adequate for aerosol particles smaller than 0.3μm?
- Is the filter efficiency for solid aerosol particles of various densities as good as the tested efficiency for liquid DOP aerosol?
- What are the effects of high temperature on filter performance?
- Do electrostatic charges on the particles and/or filter reduce filter efficiency?

Several of the more important properties of filters are described below:

- Efficiency is a measure of the ability of a filter to remove particles from an air stream. Initial efficiencies of new filters are most commonly quoted, but efficiency as a function of filter loading is equally important. Performance is frequently expressed in terms of penetration, which is a measure of the ability of particles to pass through a filter. Percent efficiency equals 100% minus percent penetration.

- Flow resistance is measured by the static pressure drop across a filter at a given flow rate.

- Particle collection capacity or filter loading is the amount of particulate that a filter can hold before flow resistance or penetration becomes excessive.

The filtration performance of these filters depends on filter composition, particle size, particle loading, and many other factors. However, the following supplier's data on MSA 1106BH with 0.3μm diameter dioctyl phthalate (DOP) liquid aerosol is typical:

Velocity (ft/min)	Initial Efficiency (%)	Flow Resistance (in. H_2O)
10	99.995	1.58 to 1.58
28	99.98	3.94 to 4.33

Linear filtration velocities in the EPA sampling train at 1 CFM are approximately as follows:

Filter Diameter (in.)	Velocity (ft/min)
2-1/4	36
3	24
4	12

EFFECT OF VELOCITY AND PARTICLE SIZE ON FILTRATION PERFORMANCE

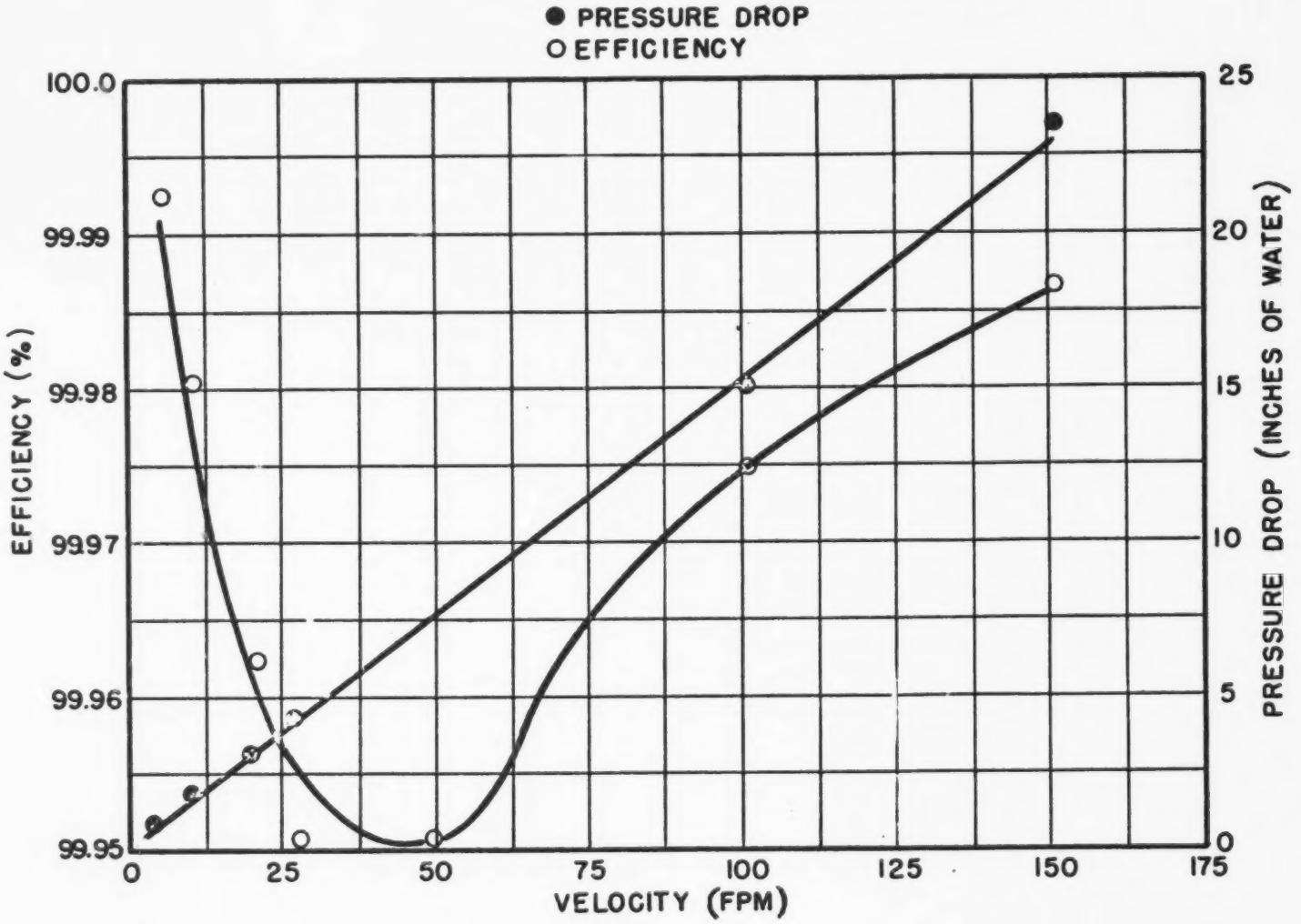
Figure 1 shows that flow resistance increases linearly with velocity. Figure 1 also shows that efficiency decreases with velocity up to about 50 ft/min and then increases. The minimum efficiency point results from the combined effects of particle diffusion and impaction, and varies with filter construction (usually between 30 to 70 ft/min).

The efficiency on particles other than 0.3μm has been the subject of much misunderstanding and controversy. It is commonly thought that the filter collects particles only down to 0.3μm diameter. This misunderstanding results from the common practice of stating 0.3μm DOP efficiencies without interpretation. The DOP test was designed during World War II to test high-efficiency gas mask filters against what was thought at that time to be the most penetrating particle size (0.3μm). The most penetrating particle size of these filters is now believed to be between 0.1 and 0.3μm diameter; the peak penetration is about 2 to 3 times the DOP penetration.[5,6,7,17] Particles smaller and larger than the most penetrating are collected with greater efficiency, largely by diffusion and impaction, respectively.

FIGURE 1

EFFICIENCY OF ABSOLUTE FILTER VS. FLOW RATE

(0.3-Micron Dioctyl Phthalate Aerosol)



Source: Doyle, et al (Ref. 11)

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EFFECT OF SOLID VS. LIQUID AEROSOLS ON FILTER EFFICIENCY

The efficiency of filters for solid vs. liquid aerosols is also not well understood. However, there is reasonable agreement that HEPA filters have equal initial efficiency for both solid and liquid aerosols.[8-10]

FILTER LOADING EFFECTS

As the filter becomes loaded with solid particles, efficiency increases, whereas the reverse is true of liquid particles. Loading with liquid particles is not a common problem with stack sampling. However, where it does occur, HEPA filters should be limited to particulate loadings of about 20 mg/sq cm of surface area.[11] The use of two or more filters in series will increase loading capacity but will also increase flow resistance.

EFFECT OF PARTICLE DENSITY ON FILTER EFFICIENCY

The effect of increased particle density on filter efficiency is to increase efficiency at high velocities, due to impaction. Reduced efficiencies with heavier particles have been measured at low velocities, but not understood.[12]

EFFECT OF ELEVATED TEMPERATURE ON FILTER PERFORMANCE

This subject is discussed by Dyment[13] as follows:

"At elevated temperature, gas viscosity will increase and therefore increased pressure drops will be obtained for the laminar flow conditions which prevail in high-efficiency paper media under normal ambient conditions.

"The effect of temperature on fibre filtration mechanisms has been summarised by Thring and Strauss[14] in terms of inertial, interception and diffusional collection. Because of the way in which the three mechanisms vary with conditions (i.e., fibre and particle sizes, fluid velocity) a general rule cannot be formulated for the effects of temperature on filtration performance. When the predominant mechanism is known, however, the effects can be predicted qualitatively. Inertia and interception efficiency are reduced by increased temperature. For the larger particles for which inertial impaction is more important, changes in particle/fibre adhesion at high temperature may exaggerate or modify this effect. The diffusion mechanism is of increasing importance as particle size diminishes below one micron. For diffusion the effect of temperature rise is to increase the fibre collection efficiency particularly for submicron particles."

Dyment also reported the results of some glass fiber filter tests with sodium chloride aerosol at temperatures up to 500°C. He found that

glass paper shrinks at these temperatures so he preheated the samples to avoid cracking failure. His data, while extremely limited, suggest that increasing temperatures to 400°C slightly decreased filter efficiency. His conclusions were as follows:

"In practice the effect of temperature and pressure on filtration mechanism and performance has not been found a determining factor in the application of high efficiency filters. The main problems are the physical and chemical effects of a high temperature environment on the materials of construction of the filter, which are manifested by reduced mechanical strength and resilience or loss of adhesion, leading to mechanical leakage and loss in efficiency."

More recent high-temperature filter efficiency tests were reported by First.[15] Heat-shrunk quartz fiber filters were tested at temperatures up to 950°F with sodium chloride aerosols of 0.14μm mass median diameter at velocities of 15 to 30 cm/sec. No effect of temperature on efficiency was found.

ELECTROSTATIC EFFECTS ON FILTER EFFICIENCY

The effects of electrostatic charges on filtration efficiency are poorly understood. The most commonly reported effects are relatively small increases in efficiency when the particles are charged, and greater increases when the filter is charged.[16,17]

The high-efficiency glass fiber filter is reported to collect at least 99% of charged particles of all sizes in the atmosphere.[18,19] Adverse electrostatic effects on filter efficiency may be possible, but appear unlikely.

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7. Author(s) A. L. Benson, P. L. Levins, A. A. Massucco, and J. R. Valentine			8. Performing Organization Rept. No.
9. Performing Organization Name and Address Arthur D. Little, Inc. Acorn Park Cambridge, Massachusetts 02140			10. Project/Task/Work Unit No. 11. Contract/Grant No. 68-02-0585
12. Sponsoring Organization Name and Address OFFICE OF RESEARCH AND DEVELOPMENT U. S. ENVIRONMENTAL PROTECTION AGENCY WASHINGTON, D. C. 20460			13. Type of Report & Period Covered 14.
15. Supplementary Notes			

16. Abstracts The purpose of this program has been to develop and characterize a high-purity filter medium for measuring particulates in stack gases at 230 to 540°C. Sufficient purity to allow optimum use of the detection limits of atomic absorption (AAS) and flame emission spectrometry (FES), and reproducible collection efficiency of 99% for dense particles as small as 0.05 um were primary objectives. The work has included analysis and purification of filter materials, filter handsheet preparation, and filter characterization. Johns-Manville Co. 99.2% silica fibers have been made into handsheet filters with satisfactory efficiency, temperature resistance, cost (about $1/ft^2), strength (about 1 lb/in.), and flexibility. The strength and flexibility result from a novel combination alkaline and 800°C heat treatment. The heat treatment also decreases extractable impurities and moisture sensitivity. The application of this treatment to machine production of filter media should be evaluated. Almost all purity requirements for optimum AAS and FES analysis have been achieved. Collection efficiency studies have been made and the results were reported. The strengthened filters are insensitive to humidity, insoluble in most acids and organic solvents, and slightly alkaline.

17. Key Words and Document Analysis. 17a. Descriptors

Air pollution
Filter materials
Efficiency
Development
Flue gases
Glass filaments
Impurities
Evaluation
Cost
Strength
Extraction
Temperature
Solubility
Purification
Flexability
Chemical analysis
Flow resistance
Humidity
Lead
Iron
Aluminum
Chrominum
Titanium
Zinc

17b. Identifiers/Open-Ended Terms

Particulates
Minimum detectable concentration
Silica fiber filters

17c. COSATI Field/Group 13B

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